Leybold

Fundamentals of Vacuum Technology

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Preface

Leybold GmbH being part of the globally active industrial Atlas Copco Group has developed into the world market leader in the area of vacuum technology. In this leading position we do not only recognize this as a task and a challenge but also as a responsibility with respect to our customers.

This brochure shall independently of the current range of products provide an easy to read overview covering the entire area of vacuum technology. The presented diagrams and data of the products shall above all promote a deeper understanding of the underlying technological functions and are not to be understood as a warranty of product properties.

The content has been enhanced through the addition of more physical principles and a number of other topics, since in the time after publication of the last edition there were naturally quite a few new developments which had to be taken into account.

To us, partnership-like customer relationships are a fundamental component of our corporate culture as are our continual investments in research and development serving as the foundation for new technological standards and innovations. In the course of our since 1850 corporate history we have in the area of vacuum technology accumulated a comprehensive process and application know-how. Jointly with our customers and partners we plan also in future to open up further markets, implement new ideas and develop pioneering products.

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Fundamentals of Vacuum Technology

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1. Quantities, their symbols, units of measure and definitions

(cf. DIN 28 400, Part 1, 1990, DIN 1314 and DIN 28 402)

1.1 Basic terms and concepts in vacuum technology

Pressure p (mbar)
of fluids (gases and liquids). (Quantity: pressure; symbol: p; unit of measure: millibar; abbreviation: mbar.) Pressure is defined in DIN Standard 1314 as the quotient of standardized force applied to a surface and the extent of this surface (force referenced to the surface area). Even though the Torr is no longer used as a unit for measuring pressure (see Section 10), it is nonetheless useful in the interest of "transparency" to mention this pressure unit: 1 Torr is that gas pressure which is able to raise a column of mercury by 1 mm at 0 °C. (Standard atmospheric pressure is 760 Torr or 760 mm Hg.) Pressure p can be more closely defined by way of subscripts:

Absolute pressure p_{abs}
Absolute pressure is always specified in vacuum technology so that the "abs" index can normally be omitted.

Total pressure p_t
The total pressure in a vessel is the sum of the partial pressures for all the gases and vapors within the vessel.

Partial pressure p_i
The partial pressure of a certain gas or vapor is the pressure which that gas or vapor would exert if it alone were present in the vessel.

Important note: Particularly in rough vacuum technology, partial pressure in a mix of gas and vapor is often understood to be the sum of the partial pressures for all the non-condensable components present in the mix – in case of the "**partial ultimate pressure**" at a rotary vane pump, for example.

Saturation vapor pressure p_s
The pressure of the saturated vapor is referred to as saturation vapor pressure p_s. p_s will be a function of temperature for any given substance.

Vapor pressure p_d
Partial pressure of those vapors which can be liquefied at the temperature of liquid nitrogen (LN_2).

Standard pressure p_n
Standard pressure p_n is defined in DIN 1343 as a pressure of p_n = 1013.25 mbar.

Ultimate pressure p_{end}
The lowest pressure which can be achieved in a vacuum vessel. The socalled ultimate pressure p_{end} depends not only on the pump's suction speed but also upon the vapor pressure p_d for the lubricants, sealants and propellants used in the pump. If a container is evacuated simply with an oil-sealed rotary (positive displacement) vacuum pump, then the ultimate pressure which can be attained will be determined primarily by the vapor pressure of the pump oil being used and, depending on the cleanliness of the vessel, also on the vapors released from the vessel walls and, of course, on the leak tightness of the vacuum vessel itself.

Ambient pressure p_{amb}
or **(absolute) atmospheric pressure**

Overpressure p_e or gauge pressure
(Index symbol from "excess")

$$p_e = p_{abs} - p_{amb}$$

Here positive values for p_e will indicate overpressure or gauge pressure; negative values will characterize a vacuum.

Working pressure p_W
During evacuation the gases and/or vapors are removed from a vessel. Gases are understood to be matter in a gaseous state which will not, however, condense at working or operating temperature. Vapor is also matter in a gaseous state but it may be liquefied at prevailing temperatures by increasing pressure. Finally, saturated vapor is matter which at the prevailing temperature is gas in equilibrium with the liquid phase of the same substance. A strict differentiation between gases and vapors will be made in the comments which follow only where necessary for complete understanding.

Particle number density n (cm^{-3})
According to the kinetic gas theory the number n of the gas molecules, referenced to the volume, is dependent on pressure p and thermodynamic temperature T as expressed in the following:

$$p = n \cdot k \cdot T \qquad (1.1)$$

n = particle number density
k = Boltzmann's constant

At a certain temperature, therefore, the pressure exerted by a gas depends only on the particle number density and not on the nature of the gas. The nature of a gaseous particle is characterized, among other factors, by its mass m_T.

Gas density ρ ($kg \cdot m^{-3}$, $g \cdot cm^{-3}$)
The product of the particle number density n and the particle mass m_T is the gas density ρ:

$$\rho = n \cdot m_T \qquad (1.2)$$

The ideal gas law
The relationship between the mass m_T of a gas molecule and the molar mass M of this gas is as follows:

$$M = N_A \cdot m_T \qquad (1.3)$$

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Avogadro's number (or constant) N_A indicates how many gas particles will be contained in a mole of gas. In addition to this, it is the proportionality factor between the gas constant R and Boltzmann's constant k:

$$R = N_A \cdot k \qquad (1.4)$$

Derivable directly from the above equations (1.1) to (1.4) is the correlation between the pressure p and the gas density ρ of an ideal gas.

$$p = \rho \cdot \frac{R \cdot T}{M} \qquad (1.5)$$

In practice we will often consider a certain enclosed volume V in which the gas is present at a certain pressure p. If m is the mass of the gas present within that volume, then

$$\rho = \frac{m}{V} \qquad (1.6)$$

The ideal gas law then follows directly from equation (1.5):

$$p \cdot V = \frac{m}{M} \cdot R \cdot T = \nu \cdot R \cdot T \qquad (1.7)$$

Here the quotient m / M is the number of moles υ present in volume V.

The simpler form applies for m / M = 1, i.e. for 1 mole:

$$p \cdot V = R \cdot T \qquad (1.7a)$$

The following numerical example is intended to illustrate the correlation between the mass of the gas and pressure for gases with differing molar masses, drawing here on the numerical values in Table IV (Chapter 9). Contained in a 10-liter volume, at 20 °C, will be

a) 1g of helium
b) 1g of nitrogen

When using the equation (1.7) there results then at V = 10 l , m = 1 g,

$$p = \frac{1 \cdot g \cdot 83.14 \cdot mbar \cdot \ell \cdot mol^{-1} \cdot K^{-1} \cdot 293 \cdot K}{10 \cdot \ell \cdot K \cdot 4 \cdot g \cdot mol^{-1}} =$$
$$= 609\ mbar$$

R = 83.14 mbar · l · mol^{-1} · K^{-1}, T = 293 K (20 °C)

$$p = \frac{1 \cdot g \cdot 83.14 \cdot mbar \cdot \ell \cdot mol^{-1} \cdot K^{-1} \cdot 293 \cdot K}{10 \cdot \ell \cdot K \cdot 28 \cdot g \cdot mol^{-1}} =$$
$$= 87\ mbar$$

In case a) where M = 4 g · $mole^{-1}$ (monatomic gas):

In case b), with M = 28 ≠ g $mole^{-1}$ (diatomic gas):

The result, though appearing to be paradoxical, is that a certain mass of a light gas exerts a greater pressure than the same mass of a heavier gas. If one takes into account, however, that at the same gas density (see Equation 1.2) more particles of a lighter gas (large n, small m) will be present than for the heavier gas (small n, large m), the results become more understandable since only the particle number density n is determinant for the pressure level, assuming equal temperature (see Equation 1.1).

The main task of vacuum technology is to reduce the particle number density n inside a given volume V. At constant temperature this is always equivalent to reducing the gas pressure p. Explicit attention must at this point be drawn to the fact that a reduction in pressure (maintaining the volume) can be achieved not only by reducing the particle number density n but also (in accordance with Equation 1.5) by reducing temperature T at constant gas density. This important phenomenon will always have to be taken into account where the temperature is not uniform throughout volume V.

The following important terms and concepts are often used in vacuum technology:

Volume V (l, m^3, cm^3)
The term volume is used to designate

a) the purely geometric, usually predetermined, volumetric content of a vacuum chamber or a complete vacuum system including all the piping and connecting spaces (this volume can be calculated);
b) the pressure-dependent volume of a gas or vapor which, for example, is moved by a pump or absorbed by an adsorption agent.

Volumetric flow (flow volume) q_v
(l/s, m^3/h, cm^3/s)
The term "flow volume" designates the volume of the gas which flows through a piping element within a unit of time, at the pressure and temperature prevailing at the particular moment. Here one must realize that, although volumetric flow may be identical, the number of molecules moved may differ, depending on the pressure and temperature.

Pumping speed S (l/s, m^3/h, cm^3/s)
The pumping speed is the volumetric flow through the pump's intake port.

$$S = \frac{dV}{dt} \qquad (1.8a)$$

If S remains constant during the pumping process, then one can use the difference quotient instead of the differential quotient:

$$S = \frac{\Delta V}{\Delta t} \qquad (1.8b)$$

(A conversion table for the various units of measure used in conjunction with pumping speed is provided in Section 9, Table VI).

Quantity of gas (pV value), (mbar · l)
The quantity of a gas can be indicated by way of its mass or its weight in the units of measure normally used for mass or weight. In practice, however, the product of p · V is often more interesting in vacuum technology than the mass or weight of a quantity of gas. The value embraces an energy dimension and is specified in millibar · liters (mbar · l) (Equation 1.7). Where the nature of the gas and its temperature are known, it is possible to use Equation 1.7b to calculate the mass m for the quantity of gas on the basis of the product of p · V:

$$p \cdot V = \frac{m}{M} \cdot R \cdot T \qquad (1.7)$$

$$m = \frac{p \cdot V \cdot M}{R \cdot T} \qquad (1.7b)$$

Although it is not absolutely correct, reference is often made in practice to the "quantity of gas" $p \cdot V$ for a certain gas. This specification is incomplete; the temperature of the gas T, usually room temperature (293 K), is normally implicitly assumed to be known.

Example: The mass of 100 mbar · l of nitrogen (N_2) at room temperature (approx. 300 K) is:

$$m = \frac{100\,\text{mbar} \cdot \ell \cdot 28\,\text{g} \cdot \text{mol}^{-1}}{83\,\text{mbar} \cdot \ell \cdot \text{mol}^{-1} \cdot \text{K}^{-1} \cdot 300\,\text{K}} =$$

$$= \frac{2800}{300 \cdot 83}\,\text{g} = 0.113\,\text{g}$$

Analogous to this, at T = 300 K:

1 mbar · l O_2 = 1.28 · 10^{-3} g O_2

70 mbar · l Ar = 1.31 · 10^{-1} g Ar

The quantity of gas flowing through a piping element during a unit of time – in accordance with the two concepts for gas quantity described above – can be indicated in either of two ways, these being:

Mass flow q_m (kg/h, g/s),
this is the quantity of a gas which flows through a piping element, referenced to time

$$q_m = \frac{m}{t} \qquad \text{or as}$$

pV flow q_{pV} (mbar · l · s^{-1}).
pV flow is the product of the pressure and volume of a quantity of gas flowing through a piping element, divided by time, i.e.:

$$q_{pV} = \frac{p \cdot V}{t} = \frac{d\,(p \cdot V)}{dt}$$

pV flow is a measure of the mass flow of the gas; the temperature to be indicated here.

Pump throughput q_{pV}
The pumping capacity (throughput) for a pump is equal either to the mass flow through the pump intake port:

$$q_m = \frac{m}{t} \qquad (1.9)$$

or to the pV flow through the pump's intake port:

$$q_{pV} = \frac{p \cdot V}{t}$$

It is normally specified in mbar · l · s^{-1}. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation

$$q_{pV} = p \cdot S \qquad (1.10a)$$

where S is the pumping speed of the pump at intake pressure of p.

(The throughput of a pump is often indicated with Q, as well.)

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in mbar ≠ l/s; the pumping speed is the "transportation capacity" which the pump makes available within a specific unit of time, measured in m^3/h or l/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to "take off" the gas moved by the high vacuum pump (see Section 2.32).

Conductance C (l · s^{-1})
The pV flow through any desired piping element, i.e. pipe or hose, valves, nozzles, openings in a wall between two vessels, etc., is indicated with

$$q_{pV} = C(p_1 - p_2) = \Delta p \cdot C \qquad (1.11)$$

Here $\Delta p = (p_1 - p_2)$ is the differential between the pressures at the inlet and outlet ends of the piping element. The proportionality factor C is designated as the conductance value or simply "conductance". It is affected by the geometry of the piping element and can even be calculated for some simpler configurations (see Section 1.5).

In the high and ultrahigh vacuum ranges, C is a constant which is independent of pressure; in the rough and medium-high regimes it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping elements must be carried out separately for the individual pressure ranges (see Section 1.5 for more detailed information).

From the definition of the volumetric flow it is also possible to state that: The conductance value C is the flow volume through a piping element. The equation (1.11) could be thought of as "Ohm's law for vacuum technology", in which q_{pV} corresponds to current, Δp the voltage and C the electrical conductance value. Analogous to Ohm's law in the science of electricity, the resistance to flow

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

has been introduced as the reciprocal value to the conductance value. The equation (1.11) can then be re-written as:

$$q_{pV} = \frac{1}{R} \cdot \Delta p \qquad (1.12)$$

The following applies directly for connection in series:

$$R_\Sigma = R1 + R2 + R3 \ldots \qquad (1.13)$$

When connected in parallel, the following applies:

$$\frac{1}{R_\Sigma} = \frac{1}{R1} + \frac{1}{R2} + \frac{1}{R3} + \cdots \qquad (1.13a)$$

Vacuum physics

Leak rate q_L (mbar · l · s^{-1})
According to the definition formulated above it is easy to understand that the size of a gas leak, i.e. movement through undesired passages or "pipe" elements, will also be given in mbar · l · s^{-1}. A leak rate is often measured or indicated with atmospheric pressure prevailing on the one side of the barrier and a vacuum at the other side (p < 1 mbar). If helium (which may be used as a tracer gas, for example) is passed through the leak under exactly these conditions, then one refers to "standard helium conditions".

Outgassing (mbar · l)
The term outgassing refers to the liberation of gases and vapors from the walls of a vacuum chamber or other components on the inside of a vacuum system. This quantity of gas is also characterized by the product of p · V, where V is the volume of the vessel into which the gases are liberated, and by p, or better Δp, the increase in pressure resulting from the introduction of gases into this volume.

Outgassing rate (mbar · l · s^{-1})
This is the outgassing through a period of time, expressed in mbar · l · s^{-1}.

Outgassing rate (mbar · l · s^{-1} · cm^{-2})
(referenced to surface area)
In order to estimate the amount of gas which will have to be extracted, knowledge of the size of the interior surface area, its material and the surface characteristics, their outgassing rate referenced to the surface area and their progress through time are important.

Mean free path of the molecules λ (cm) **and collision rate z** (s^{-1})
The concept that a gas comprises a large number of distinct particles between which – aside from the collisions – there are no effective forces, has led to a number of theoretical considerations which we summarize today under the designation "kinetic theory of gases".

One of the first and at the same time most beneficial results of this theory was the calculation of gas pressure p as a function of gas density and the mean square of velocity c^2 for the individual gas molecules in the mass of molecules m_T:

$$p = \frac{1}{3}\ \rho \cdot \overline{c^2} = \frac{1}{3} \cdot n \cdot m_T \cdot \overline{c^2} \qquad (1.14)$$

where

$$\overline{c^2} = 3 \cdot \frac{k \cdot T}{m_T} \qquad (1.15)$$

The gas molecules fly about and among each other, at every possible velocity, and bombard both the vessel walls and collide (elastically) with each other. This motion of the gas molecules is described numerically with the assistance of the kinetic theory of gases. A molecule's average number of collisions over a given period of time, the so-called collision index z, and the mean path distance which each gas molecule covers between two collisions with other molecules, the so-called mean free path length λ, are described as shown below as a function of the mean molecule velocity $\bar{c}$ the molecule diameter 2r and the particle number density molecules n – as a very good approximation:

$$Z = \frac{\bar{c}}{\lambda} \qquad (1.16)$$

where $$\bar{c} = \sqrt{\frac{8 \cdot k \cdot T}{\pi \cdot m_T}} = \sqrt{\frac{8 \cdot R \cdot T}{\pi \cdot M}} \qquad (1.17)$$

and $$\lambda = \frac{1}{\pi \cdot \sqrt{2} \cdot n \cdot (2r)^2} \qquad (1.18)$$

Thus the mean free path length λ for the particle number density n is, in accordance with equation (1.1), inversely proportional to pressure p. Thus the following relationship holds, at constant temperature T, for every gas

$$\lambda \cdot p = \text{const} \qquad (1.19)$$

Used to calculate the mean free path length λ for any arbitrary pressures and various gases are Table III and Fig. 9.1 in Chapter 9. The equations in gas kinetics which are most important for vacuum technology are also summarized (Table IV) in chapter 9.

Impingement rate z_A ($cm^{-2} \cdot s^{-1}$) and
monolayer formation time τ (s)
A technique frequently used to characterize the pressure state in the high vacuum regime is the calculation of the time required to form a monomolecular or monoatomic layer on a gas-free surface, on the assumption that every molecule will stick to the surface. This monolayer formation time is closely related with the so-called impingement rate z_A. With a gas at rest the impingement rate will indicate the number of molecules which collide with the surface inside the vacuum vessel per unit of time and surface area:

$$z_A = \frac{n \cdot \bar{c}}{4} \qquad (1.20)$$

If a is the number of spaces, per unit of surface area, which can accept a specific gas, then the monolayer formation time is

$$\tau = \frac{a}{z_A} = \frac{4 \cdot a}{n \cdot \bar{c}} \qquad (1.21)$$

Collision frequency z_v ($cm^{-3} \cdot s^{-1}$)
This is the product of the collision rate z and the **half** of the particle number density n, since the collision of **two** molecules is to be counted as only **one** collision:

$$z_V = \frac{n}{2} \cdot z \qquad (1.21a)$$

1.2 Atmospheric air

Prior to evacuation, every vacuum system on earth contains air and it will always be surrounded by air during operation. This makes it necessary to be familiar with the physical and chemical properties of atmospheric air.

The atmosphere is made up of a number of gases and, near the earth's surface, water vapor as well. The pressure exerted by atmospheric air is referenced to sea level. Average atmospheric pressure is 1013 mbar (equivalent to the "atmosphere", a unit of measure used earlier). Table VIII in Chapter 9 shows the composition of the standard atmosphere at relative humidity of 50 % and temperature of 20 °C. In terms of vacuum technology the following points should be noted in regard to the composition of the air:

a) The water vapor contained in the air, varying according to the humidity level, plays an important part when evacuating a vacuum plant (see Section 2.2.3).

b) The considerable amount of the inert gas argon should be taken into account in evacuation procedures using sorption pumps (see Section 2.1.8).

c) In spite of the very low content of helium in the atmosphere, only about 5 ppm (parts per million), this inert gas makes itself particularly obvious in ultrahigh vacuum systems which are sealed with Viton or which incorporate glass or quartz components. Helium is able to permeate these substances to a measurable extent.

The pressure of atmospheric air falls with rising altitude above the earth's surface (see Fig. 9.3 in Chapter 9). High vacuum prevails at an altitude of about 100 km and ultrahigh vacuum above 400 km. The composition of the air also changes with the distance to the surface of the earth (see Fig. 9.4 in Chapter 9).

1.3 Gas laws and models

1.3.1 Continuum theory

Model concept: Gas is "pourable" (fluid) and flows in a way similar to a liquid. The continuum theory and the summarization of the gas laws which follows are based on experience and can explain all the processes in gases near atmospheric pressure. Only after it became possible using ever better vacuum pumps to dilute the air to the extent that the mean free path rose far beyond the dimensions of the vessel were more far-reaching assumptions necessary; these culminated in the kinetic gas theory. The kinetic gas theory applies throughout the entire pressure range; the continuum theory represents the (historically older) special case in the gas laws where atmospheric conditions prevail.

Summary of the most important gas laws (continuum theory)

Boyle-Mariotte Law

$$p \cdot V = \text{const.}$$

for T = constant (isotherm)

Gay-Lussac's Law (Charles' Law)

$$V = V_0(1 + \beta \cdot t)$$

for p = constant (isobar)

Amonton's Law

$$p = p_0(1 + \gamma \cdot t)$$

for V = constant (isochor)

Dalton's Law

$$\sum_i p_i = p_{total}$$

Poisson's Law

$$p \cdot V^{\kappa} = \text{const}$$

(adiabatic)

Avogadro's Law

$$\frac{m_1}{V_1} : \frac{m_2}{V_2} = M_1 : M_2$$

Ideal gas Law

$$p \cdot V = \frac{m}{M} \cdot R \cdot T = \nu \cdot R \cdot T$$

Also: **Equation of state for ideal gases** (from the continuum theory)

van der Waals' Equation

$$(p + \frac{a}{V_m^2}) \cdot (V_m - b) = R \cdot T$$

a, b = constants (internal pressure, covolumes)
Vm = Molar volume
also: **Equation of state for real gases**

Clausius-Clapeyron Equation

$$L = T \cdot \frac{dp}{dT} \cdot (V_{m,v} - V_{m,l})$$

L = Enthalpy of evaporation,
T = Evaporation temperature,
$V_{m,v}$, $V_{m,l}$ = Molar volumes of vapor or liquid

1.3.2 Kinetic gas theory

With the acceptance of the atomic view of the world – accompanied by the necessity to explain reactions in extremely dilute gases (where the continuum theory fails) – the "kinetic gas theory" was developed. Using this it is possible not only to derive the ideal gas law in another manner but also to calculate many other quantities involved with the kinetics of gases – such as collision rates, mean free path lengths, monolayer formation time, diffusion constants and many other quantities.

Vacuum physics

Model concepts and basic assumptions:

1. Atoms/molecules are points.
2. Forces are transmitted from one to another only by collision.
3. The collisions are elastic.
4. Molecular disorder (randomness) prevails.

A very much simplified model was developed by Krönig. Located in a cube are N particles, one-sixth of which are moving toward any given surface of the cube. If the edge of the cube is 1 cm long, then it will contain n particles (particle number density); within a unit of time $n \cdot c \cdot \Delta t/6$ molecules will reach each wall where the change of pulse per molecule, due to the change of direction through 180 °, will be equal to $2 \cdot m_T \cdot c$. The sum of the pulse changes for all the molecules impinging on the wall will result in a force effective on this wall or the pressure acting on the wall, per unit of surface area.

$$\frac{n}{6} \cdot c \cdot 2 \cdot m_T \cdot c = \frac{1}{3} \cdot n \cdot c^2 \cdot m_T = p$$

where $n = \frac{N}{V}$

Derived from this is $p \cdot V = \frac{1}{3} \cdot N \cdot m_T \cdot c^2$

Ideal gas law (derived from the kinetic gas theory)

If one replaces c^2 with $\overline{c^2}$ then a comparison of these two "general" gas equations will show:

$$p \cdot V = N \cdot \left(\frac{m_T \cdot R}{M}\right) \cdot T = \frac{2}{3} \cdot N \cdot \left(\frac{m_T \cdot \overline{c^2}}{2}\right) \quad \text{or}$$

The expression in brackets on the left-hand side is the Boltzmann constant k; that on the right-hand side a measure of the molecules' mean kinetic energy:

Boltzmann constant

$$k = \frac{m_T \cdot R}{M} = 1.38 \cdot 10^{-23} \frac{J}{K}$$

Mean kinetic energy of the molecules

$$\overline{E_{kin}} = \frac{m_T \cdot \overline{c^2}}{2}$$

thus $p \cdot V = N \cdot k \cdot T = \frac{2}{3} \cdot N \cdot \overline{E_{kin}}$

In this form the gas equation provides a gas-kinetic indication of the temperature!

The mass of the molecules is

$$m_T = \frac{M}{N_A} = \frac{\text{Mass / mol}}{\text{Molecules / mol}}$$

where N_A is Avogadro's number (previously: Loschmidt number).

Avogadro constant

$N_A = 6.022 \cdot 10^{23}\ mol^{-1}$

For 1 mole, $\frac{m_T}{M} = 1$ and

V = Vm = 22.414 l (molar volume);

Thus from the ideal gas law at standard conditions (T_n = 273.15 K and p_n = 1013.25 mbar):

$$p \cdot V = \frac{m}{M} \cdot R \cdot T$$

For the general gas constant:

$$R = \frac{1013.25\ \text{mbar} \cdot 22.4\,\ell \cdot \text{mol}^{-1}}{273.15\ \text{K}} =$$

$$= 83.14 \frac{\text{mbar} \cdot \ell}{\text{mol} \cdot \text{K}}$$

1.4 The pressure ranges in vacuum technologyand their characterization

(See also Table IX in Chapter 9.) It is common in vacuum technology to subdivide its wide overall pressure range – which spans more than 16 powers of ten – into smaller individual regimes. These are generally defined as follows:

Rough vacuum (RV)	1000 – 1	mbar
Medium vacuum (MV)	1 – 10^{-3}	mbar
High vacuum (HV)	10^{-3} – 10^{-7}	mbar
Ultrahigh vacuum (UHV)	10^{-7} – (10^{-14})	mbar

This division is, naturally, somewhat arbitrary. Chemists in particular may refer to the spectrum of greatest interest to them, lying between 100 and 1 mbar, as "intermediate vacuum". Some engineers may not refer to vacuum at all but instead speak of "low pressure" or even "negative pressure". The pressure regimes listed above can, however, be delineated quite satisfactorily from an observation of the gas-kinetic situation and the nature of gas flow. The operating technologies in the various ranges will differ, as well.

1.5 Types of flow and conductance

Three types of flow are mainly encountered in vacuum technology: viscous or continuous flow, molecular flow and – at the transition between these two – the so-called Knudsen flow.

1.5.1 Types of flow

Viscous or continuum flow
This will be found almost exclusively in the rough vacuum range. The character of this type of flow is determined by the interaction of the molecules. Consequently internal friction, the viscosity of the flowing substance, is a major factor. If vortex motion appears in the streaming process, one speaks of **turbulent flow**. If various layers of the flowing medium slide one over the other, then the term laminar flow or layer flux may be applied.

Laminar flow in circular tubes with parabolic velocity distribution is known as **Poiseuille flow**. This special case is found frequently in vacuum technology. Viscous flow will generally be found where the molecules' mean free path is considerably shorter than the diameter of the pipe: $\lambda \ll d$.

A characteristic quantity describing the viscous flow state is the dimensionless Reynolds number Re.

Re is the product of the pipe diameter, flow velocity, density and reciprocal value of the viscosity (internal friction) of the gas which is flowing. Flow is turbulent where Re > 2200, laminar where Re < 2200.

The phenomenon of **choked flow** may also be observed in the viscous flow situation. It plays a part when venting and evacuating a vacuum vessel and where there are leaks.

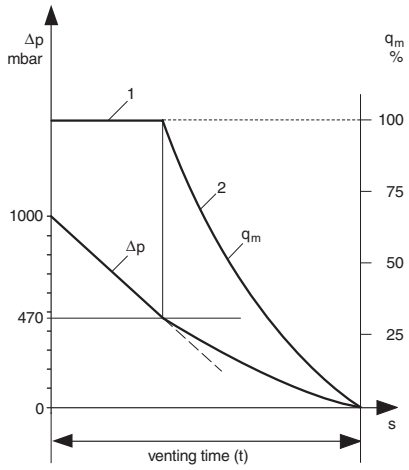
Gas will always flow where there is a difference in pressure $\Delta p = (p_1 - p_2) > 0$. The intensity of the gas flow, i.e. the quantity of gas flowing over a period of time, rises with the pressure differential. In the case of viscous flow, however, this will be the case only until the flow velocity, which also rises, reaches the speed of sound. This is always the case at a certain pressure differential and this value may be characterized as "critical":

$$\Delta p_{crit} = p_1 \left[1 - \left(\frac{p_2}{p_1}\right)_{crit}\right] \qquad (1.22)$$

A further rise in $\Delta p > \Delta p_{crit}$ would not result in any further rise in gas flow; any increase is inhibited. For air at 20,°C the gas dynamics theory reveals a critical value of

$$\left(\frac{p_2}{p_1}\right)_{crit} = 0.528 \qquad (1.23)$$

The chart in Fig. 1.1 represents schematically the venting (or airing) of an evacuated container through an opening in the envelope (venting valve), allowing ambient air at p = 1000 mbar to enter. In accordance with the information given above, the resultant critical pressure is $\Delta p_{crit} = 1000 \cdot (1 - 0.528)$ mbar ≈ 470 mbar; i.e. where $\Delta p > 470$ mbar the flow rate will be choked; where $\Delta p < 470$ mbar the gas flow will decline.



1 – Gas flow rate qm choked = constant (maximum value)
2 – Gas flow not impeded, qm drops to Δp = 0

Fig. 1.1 Schematic representation of venting an evacuated vessel

Molecular flow
Molecular flow prevails in the high and ultrahigh vacuum ranges. In these regimes the molecules can move freely, without any mutual interference. Molecular flow is present where the mean free path length for a particle is very much larger than the diameter of the pipe: $\lambda \gg d$.

Knudsen flow
The transitional range between viscous flow and molecular flow is known as Knudsen flow. It is prevalent in the medium vacuum range: $\lambda \approx d$.

The product of pressure p and pipe diameter d for a particular gas at a certain temperature can serve as a characterizing quantity for the various types of flow. Using the numerical values provided in Table III, Chapter 9, the following equivalent relationships exist for air at 20 °C:

Rough vacuum – Viscous flow

$$\lambda < \frac{d}{100} \quad \Leftrightarrow \quad p \cdot d > 6.0 \cdot 10^{-1} \text{ mbar} \cdot \text{cm}$$

Medium vacuum – Knudsen flow

$$\frac{d}{100} < \lambda < \frac{d}{2} \quad \Leftrightarrow$$

$$\Leftrightarrow \quad 6 \cdot 10^{-1} > p \cdot d > 1.3 \cdot 10^{-2} \text{ mbar} \cdot \text{cm}$$

High and ultrahigh vacuum – Molecular flow

$$\lambda > \frac{d}{2} \quad \Leftrightarrow \quad p \cdot d < 1.3 \cdot 10^{-2} \text{ mbar} \neq \text{cm}$$

In the viscous flow range the preferred speed direction for all the gas molecules will be identical to the macroscopic direction of flow for the gas. This alignment is compelled by the fact that the gas particles are densely packed and will collide with one another far more often than with the boundary walls of the apparatus. The macroscopic speed of the gas is a

"group velocity" and is not identical with the "thermal velocity" of the gas molecules.

In the molecular flow range, on the other hand, impact of the particles with the walls predominates. As a result of reflection (but also of desorption following a certain residence period on the container walls) a gas particle can move in any arbitrary direction in a high vacuum; it is no longer possible to speak of "flow" in the macroscopic sense.

It would make little sense to attempt to determine the vacuum pressure ranges as a function of the geometric operating situation in each case. The limits for the individual pressure regimes (see Table IX in Chapter 9) were selected in such a way that when working with normal-sized laboratory equipment the collisions of the gas particles among each other will predominate in the rough vacuum range whereas in the high and ultrahigh vacuum ranges impact of the gas particles on the container walls will predominate.

In the high and ultrahigh vacuum ranges the properties of the vacuum container wall will be of decisive importance since below 10^{-3} mbar there will be more gas molecules on the surfaces than in the chamber itself. If one assumes a monomolecular adsorbed layer on the inside wall of an evacuated sphere with 1 l volume, then the ratio of the number of adsorbed particles to the number of free molecules in the space will be as follows:

at 1 mbar 10^{-2}
at 10^{-6} mbar 10^{+4}
at 10^{-11} mbar 10^{+9}

For this reason the monolayer formation time τ (see Section 1.1) is used to characterize ultrahigh vacuum and to distinguish this regime from the high vacuum range. The monolayer formation time τ is only a fraction of a second in the high vacuum range while in the ultrahigh vacuum range it extends over a period of minutes or hours. Surfaces free of gases can therefore be achieved (and maintained over longer periods of time) only under ultrahigh vacuum conditions.

Further physical properties change as pressure changes. For example, the thermal conductivity and the internal friction of gases in the medium vacuum range are highly sensitive to pressure. In the rough and high vacuum regimes, in contrast, these two properties are virtually independent of pressure.

Thus, not only will the pumps needed to achieve these pressures in the various vacuum ranges differ, but also different vacuum gauges will be required. A clear arrangement of pumps and measurement instruments for the individual pressure ranges is shown in Figures 9.16 and 9.16a in Chapter 9.

1.5.2 Calculating conductance values

The effective pumping speed required to evacuate a vessel or to carry out a process inside a vacuum system will correspond to the inlet speed of a particular pump (or the pump system) only if the pump is joined directly to the vessel or system. Practically speaking, this is possible only in rare situations. It is almost always necessary to include an intermediate piping system comprising valves, separators, cold traps and the like. All this represents an resistance to flow, the consequence of which is that the effective pumping speed S_{eff} is always less than the pumping speed S of the pump or the pumping system alone. Thus to ensure a certain effective pumping speed at the vacuum vessel it is necessary to select a pump with greater pumping speed. The correlation between S and S_{eff} is indicated by the following basic equation:

$$\frac{1}{S_{eff}} = \frac{1}{S} + \frac{1}{C} \qquad (1.24)$$

Here C is the total conductance value for the pipe system, made up of the individual values for the various components which are connected in series (valves, baffles, separators, etc.):

$$\frac{1}{C} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \ldots \frac{1}{C_n} \qquad (1.25)$$

Equation (1.24) tells us that only in the situation where $C = \infty$ (meaning that the flow resistance is equal to 0) will $S = S_{eff}$. A number of helpful equations is available to the vacuum technologist for calculating the conductance value C for piping sections. The conductance values for valves, cold traps, separators and vapor barriers will, as a rule, have to be determined empirically.

It should be noted that in general that the conductance in a vacuum component is not a constant value which is independent of prevailing vacuum levels, but rather depends strongly on the nature of the flow (continuum or molecular flow; see below) and thus on pressure. When using conductance indices in vacuum technology calculations, therefore, it is always necessary to pay attention to the fact that only the conductance values applicable to a certain pressure regime may be applied in that regime.

1.5.3 Conductance for piping and orifices

Conductance values will depend not only on the pressure and the nature of the gas which is flowing, but also on the sectional shape of the conducting element (e.g. circular or elliptical cross section). Other factors are the length and whether the element is straight or curved. The result is that various equations are required to take into account practical situations. Each of these equations is valid only for a particular pressure range. This is always to be considered in calculations.

a) Conductance for a straight pipe, which is not too short, of length l, with a circular cross section of diameter d for the laminar, Knudsen and molecular flow ranges, valid for air at 20 °C (Knudsen equation):

$$C = 135\frac{d^4}{l}\bar{p} + 12.1\frac{d^3}{l}\cdot\frac{1 + 192\cdot d\cdot\bar{p}}{1 + 237\cdot d\cdot\bar{p}}\ \ell/s \qquad (1.26)$$

where

$$\bar{p} = \frac{p_1 + p_2}{2}$$

d = Pipe inside diameter in cm
l = Pipe length in cm ($l \geq 10\ d$)
p_1 = Pressure at start of pipe (along the direction of flow) in mbar
p_2 = Pressure at end of pipe (along the direction of flow) in mbar

If one rewrites the second term in (1.26) in the following form

$$C = 12.1 \cdot \frac{d^3}{l} \cdot f\,(d \cdot \bar{p}) \qquad (1.26a)$$

with

$$f\,(d \cdot \bar{p}) = \frac{1 + 203 \cdot d \cdot \bar{p} + 2.78 \cdot 10^3 \cdot d^2 \cdot \bar{p}^2}{1 + 237 \cdot d \cdot \bar{p}} \qquad (1.27)$$

it is possible to derive the two important limits from the course of the function f $(d \cdot \bar{p})$:

Limit for laminar flow
$(d \cdot \bar{p} > 6 \cdot 10^{-1}$ mbar · cm):

$$C = 135 \cdot \frac{d^4}{l} \cdot \bar{p} \quad \ell/s \qquad (1.28a)$$

Limit for molecular flow
$(d \cdot \bar{p} < 10^{-2}$ mbar · cm) :

$$C = 12.1 \cdot \frac{d^3}{l} \ \ell/s \qquad (1.28b)$$

In the molecular flow region the conductance value is independent of pressure!
The complete Knudsen equation (1.26) will have to be used in the transitional area $10^{-2} < d \cdot \bar{p} < 6 \cdot 10^{-1}$ mbar · cm. Conductance values for straight pipes of standard nominal diameters are shown in Figure 9.5 (laminar flow) and Figure 9.6 (molecular flow) in Chapter 9. Additional nomograms for conductance determination will also be found in Chapter 9 (Figures 9.8 and 9.9).

b) **Conductance value C for an orifice A**
(A in cm²): For continuum flow (viscous flow) the following equations (after Prandtl) apply to air at 20 °C where $p_2/p_1 = \delta$:

for $\delta \geq 0.528$ (1.29)

$$C_{visc} = 76.6 \cdot \delta^{0.712} \cdot \sqrt{1 - \delta^{0.288}} \cdot \frac{A}{1-\delta} \ \frac{\ell}{s}$$

for $\delta \leq 0.528$ (1.29a)

$$C_{visc} = 20 \cdot \frac{A}{1-\delta} \ \frac{\ell}{s}$$

and for $\delta \leq 0{,}03$ (1.29b)

$$C_{visc} = 20 \cdot A \ \frac{\ell}{s}$$

$\delta = 0.528$ is the critical pressure situation for air

$$\left(\frac{p_2}{p_1}\right)_{crit}$$

Flow is choked at $\delta < 0.528$; gas flow is thus constant. In the case of molecular flow (high vacuum) the following will apply for air:

$$Cmol = 11{,}6 \cdot A \cdot l \cdot s\text{-}1 \quad (A \text{ in } cm^2) \qquad (1.30)$$

Given in addition in Figure 1.3 are the pumping speeds S^*_{visc} and S^*_{mol} referenced to the area A of the opening and as a function of $\delta = p_2/p_1$. The equations given apply to air at 20 °C. The molar masses for the flowing gas are taken into consideration in the general equations, not shown here.

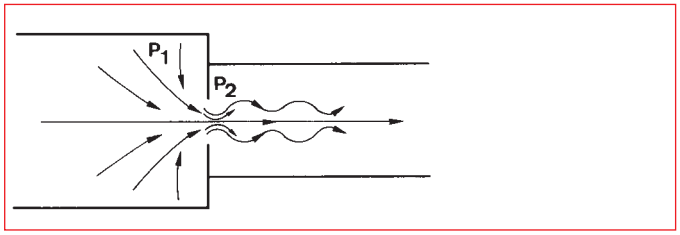


Fig. 1.2 Flow of a gas through an opening (A) at high pressures (viscous flow)

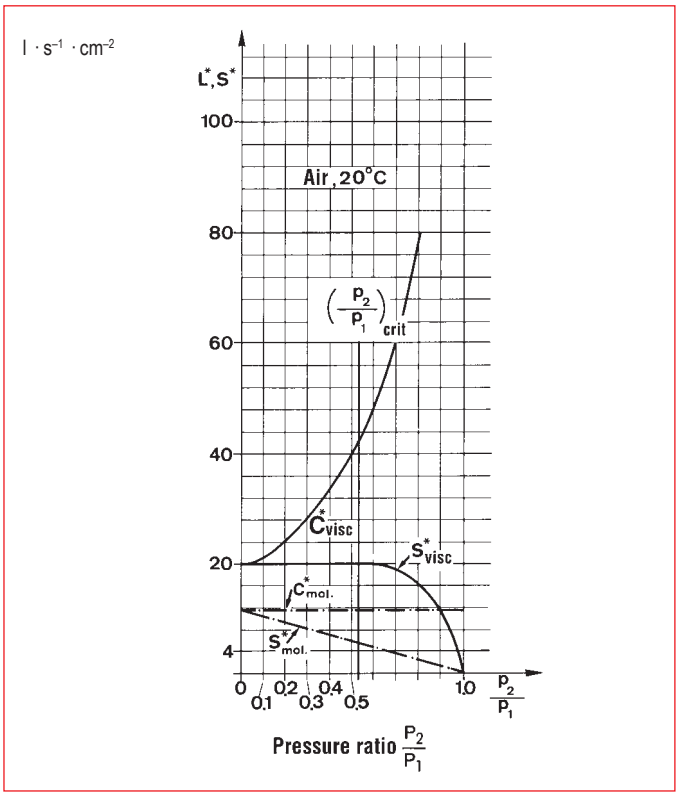


Fig. 1.3 Conductance values relative to the area, C^*_{visc}, C^*_{mol}, and pumping speed S^*_{visc} and S^*_{mol} for an orifice A, depending on the pressure relationship p_2/p_1 for air at 20 °C.

Gas (20 °C)	Molecular flow	Laminar flow
Air	1.00	1.00
Oxygen	0.947	0.91
Neon	1.013	1.05
Helium	2.64	0.92
Hydrogen	3.77	2.07
Carbon dioxide	0.808	1.26
Water vapor	1.263	1.73

Table 1.1 Conversion factors (see text)

When working with other gases it will be necessary to multiply the conductance values specified for air by the factors shown in Table 1.1.

Nomographic determination of conductance values
The conductance values for piping and openings through which air and other gases pass can be determined with nomographic methods. It is possible not only to determine the conductance value for piping at specified values for diameter, length and pressure, but also the size of the pipe diameter required when a pumping set is to achieve a certain effective pumping speed at a given pressure and given length of the line. It is also possible to establish the maximum permissible pipe length where the other parameters are known. The values obtained naturally do not apply to turbulent flows. In doubtful situations, the Reynolds number Re (see Section 1.5.) should be estimated using the relationship which is approximated below

$$Re = 15 \cdot \frac{q_{pV}}{d} \qquad (1.31)$$

Here $q_{pV} = S \cdot p$ is the flow output in mbar l/s, d the diameter of the pipe in cm.

A compilation of nomograms which have proved to be useful in practice will be found in Chapter 9.

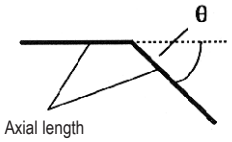
1.5.4 Conductance values for other elements

Where the line contains elbows or other curves (such as in right-angle valves), these can be taken into account by assuming a greater effective length l_{eff} of the line. This can be estimated as follows:

$$l_{eff} = l_{axial} + 1.33 \cdot \frac{\theta}{180°} \cdot d \qquad (1.32)$$

Where

l_{axial} : axial length of the line (in cm)
l_{eff} : Effective length of the line (in cm)
d : Inside diameter of the line (in cm)
θ : Angle of the elbow (degrees of angle)



The technical data in the Leybold catalog states the conductance values for vapor barriers, cold traps, adsorption traps and valves for the molecular flow range. At higher pressures, e.g. in the Knudsen and laminar flow ranges, valves will have about the same conductance values as pipes of corresponding nominal diameters and axial lengths. In regard to right-angle valves the conductance calculation for an elbow must be applied.

In the case of dust filters which are used to protect gas ballast pumps and roots pumps, the percentage restriction value for the various pressure levels are listed in the catalog. Other components, namely the condensate separators and condensers, are designed so that they will not reduce pumping speed to any appreciable extent.

The following may be used as a rule of thumb for dimensioning vacuum lines: **The lines should be as short and as wide as possible.** They must exhibit at least the same cross-section as the intake port at the pump.
If particular circumstances prevent shortening the suction line, then it is advisable, whenever this is justifiable from the engineering and economic points of view, to include a roots pump in the suction line. This then acts as a gas entrainment pump which reduces line impedance.

2. Vacuum generation

2.1. Vacuum pumps: A survey

Vacuum pumps are used to reduce the gas pressure in a certain volume and thus the gas density (see equation 1.5). Consequently consider the gas particles need to be removed from the volume. Basically differentiation is made between two classes of vacuum pumps:

a) Vacuum pumps where – via one or several compression stages – the gas particles are removed from the volume which is to be pumped and ejected into the atmosphere (compression pumps). The gas particles are pumped by means of displacement or pulse transfer.

b) Vacuum pumps where the gas particles which are to be removed condense on or are bonded by other means (e.g. chemically) to a solid surface, which often is part of the boundary forming volume itself.

A classification which is more in line with the state-of-the-art and practical applications makes a difference between the following types of pumps, of which the first three classes belong to the compression pumps and where the two remaining classes belong to the condensation and getter pumps:

1. Pumps which operate with periodically increasing and decreasing pump cham-ber volumes (rotary vane and rotary plunger pumps; also trochoid pumps)
2. Pumps which transport quantities of gas from the low pressure side to the high pressure side without changing the volume of the pumping chamber (Roots pumps, turbomolecular pumps)
3. Pumps where the pumping effect is based mainly on the diffusion of gases into a gas-free high speed vapor jet (vapor pumps)
4. Pumps which pump vapors by means of condensation (condensers) and pumps which pump permanent gases by way of condensation at very low temperatures (cryopumps)
5. Pumps which bond or incorporate gases by adsorption or absorption to surfaces which are substantially free of gases (sorption pumps).

A survey on these classes of vacuum pumps is given in the diagram of Table 2.1.

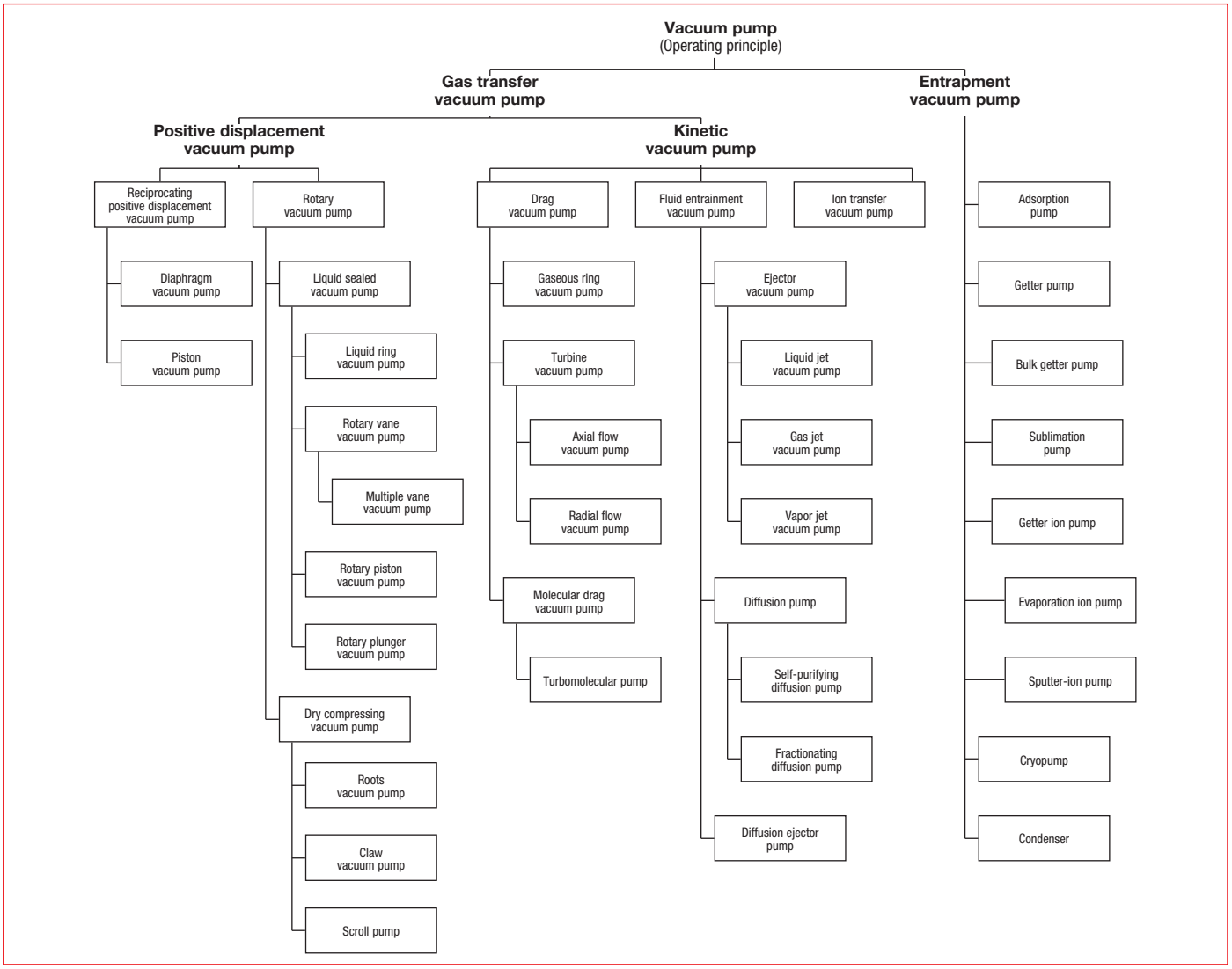


Table 2.1 Classification of vacuum pumps

Vacuum generation

2.1.1 Oscillation displacement vacuum pumps

2.1.1.1 Diaphragm pumps

Recently, diaphragm pumps have becoming ever more important, mainly for environmental reasons. They are alternatives to water jet vacuum pumps, since diaphragm pumps do not produce any waste water. Overall, a diaphragm vacuum pump can save up to 90 % of the operating costs compared to a water jet pump. Compared to rotary vane pumps, the pumping chamber of diaphragm pumps are entirely free of oil. By design, no oil immersed shaft seals are required. Diaphragm vacuum pumps are single or multi-stage dry compressing vacuum pumps (diaphragm pumps having up to four stages are being manufactured). Here the circumference of a diaphragm is tensioned between a pump head and the casing wall (Fig. 2.1). It is moved in an oscillating way by means of a connecting rod and an eccentric. The pumping or compression chamber, the volume of which increases and decreases periodically, effects the pumping action. The valves are arranged in such a way that during the phase where the volume of the pumping chamber increases it is open to the intake line. During compression, the pumping chamber is linked to the exhaust line. The diaphragm provides a hermetic seal between the gear chamber and the pumping chamber so that it remains free of oil and lubricants (dry compressing vacuum pump). Diaphragm and valves are the only components in contact with the medium which is to be pumped. When coating the diaphragm with PTFE (Teflon) and when manufacturing the inlet and exhaust valves of a highly fluorinated elastomer as in the case of the DIVAC from LEYBOLD, it is then possible to pump aggressive vapors and gases. It is thus well suited for vacuum applications in the chemistry lab.

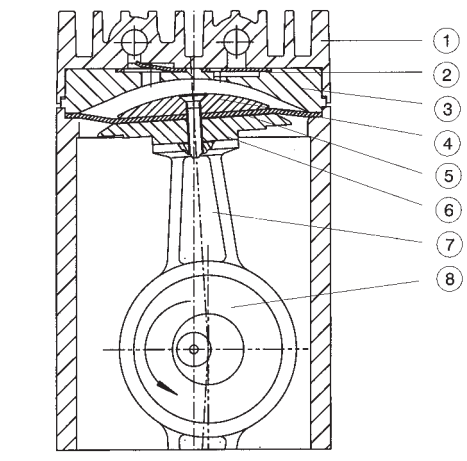
Due to the limited elastic deformability of the diaphragm only a comparatively low pumping speed is obtained. In the case of this pumping principle a volume remains at the upper dead center – the so called “dead space” – from where the gases can not be moved to the exhaust line. The quantity of gas which remains at the exhaust pressure expands into the expanding pumping chamber during the subsequent suction stroke thereby filling it, so that as the intake pressure reduces the quantity of inflowing new gas reduces more and more. Thus volumetric efficiency worsens continuously for this reason. Diaphragm vacuum pumps are not capable of attaining a higher compression ratio than the ratio between “dead space” and maximum volume of the pumping chamber. In the case of single-stage diaphragm vacuum pumps the attainable ultimate pressure amounts to approximately 80 mbar. Two-stage pumps such as the DIVAC from LEYBOLD can attain about 10 mbar (see Fig. 2.2), three-stage pumps can attain about 2 mbar and four-stage diaphragm pumps can reach about $5 \cdot 10^{-1}$ mbar.

Diaphragm pumps offering such a low ultimate pressure are suited as backing pumps for turbomolecular pumps with fully integrated Scroll stages (compound or wide range turbomolecular pumps, such as the TURBOVAC 55 from LEYBOLD). In this way a pump system is obtained which is absolutely free of oil, this being of great importance to measurement arrangements involving mass spectrometer systems and leak detectors. In contrast to rotary vane pumps this combination of pumps for leak detectors offers the advantage that naturally no helium is dissolved in the diaphragm pump thereby entirely avoiding a possible build up of a helium background.

2.1.2 Liquid sealed rotary displacement pumps

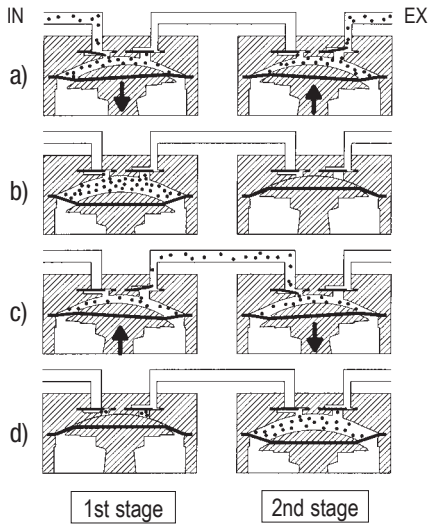
2.1.2.1 Liquid ring pumps

Due to the pumping principle and the simple design, liquid ring vacuum pumps are particularly suited to pumping gases and vapors which may also contain small amounts of liquid. Air, saturated with water vapors or other gases containing condensable constituents, may be pumped without problems. By design, liquid ring pumps are insensitive to any contamination



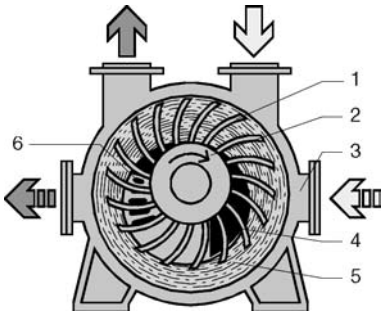
(1) Casing lid
(2) Valves
(3) Lid
(4) Diaphragm disk
(5) Diaphragm
(6) Diaphragm support disk
(7) Connecting rod
(8) Eccentric disk

Fig. 2.1 Schematic on the design of a diaphragm pump stage (Vacuubrand)



Opening and closing of the valves, path and pumping mechanism during four subsequent phases of a turn of the connecting rod (a-d)

Fig. 2.2 Principle of operation for a two-stage diaphragm pump (Vacuubrand)



1 Rotor
2 Rotor shaft
3 Casing
4 Intake channel
5 Liquid ring
6 Flex. discharge channel

Fig. 2.3 Liquid ring vacuum pump, schematic (Siemens)

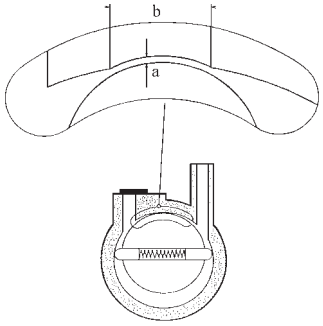
which may be present in the gas flow. The attainable intake pressures are in the region between atmospheric pressure and the vapor pressure of the operating liquid used. For water at 15 °C it is possible to attain an operating pressure of 33 mbar. A typical application of water ring vacuum pumps is venting of steam turbines in power plants. Liquid ring vacuum pumps (Fig. 2.3) are rotary displacement pumps which require an operating liquid which rotates during operation to pump the gas. The blade wheel is arranged eccentrically in a cylindrical casing. When not in operation, approximately half of the pump is filled with the operating fluid. In the axial direction the cells formed by the blade wheel are limited and sealed off by "control discs". These control discs are equipped with suction and ejection slots which lead to the corresponding ports of the pump. After having switched on such a pump the blade wheel runs eccentrically within the casing; thus a concentrically rotating liquid ring is created which at the narrowest point fully fills the space between the casing and the blade wheel and which retracts from the chambers as the rotation continues. The gas is sucked in as the chambers empty and compression is obtained by subsequent filling. The limits for the intake or discharge process are set by the geometry of the openings in the control discs.

In addition to the task of compression, the operating fluid fulfills three further important tasks:

1. Removal of the heat produced by the compression process.
2. Uptake of liquids and vapors (condensate).
3. Providing the seal between the blade wheel and the casing.

2.1.2.2 Oil sealed rotary displacement pumps

A displacement vacuum pump is generally a vacuum pump in which the gas which is to be pumped is sucked in with the aid of pistons, rotors, vanes and valves or similar, possibly compressed and then discharged. The pumping process is effected by the rotary motion of the piston inside the pump. Differentiation should be made between oiled and dry compressing displacement pumps. By the use of sealing oil it is possible to attain in a single-stage high compression ratios of up to about 10^5. Without oil, "inner leakiness" is considerably greater and the attainable compression ratio is correspondingly less, about 10.



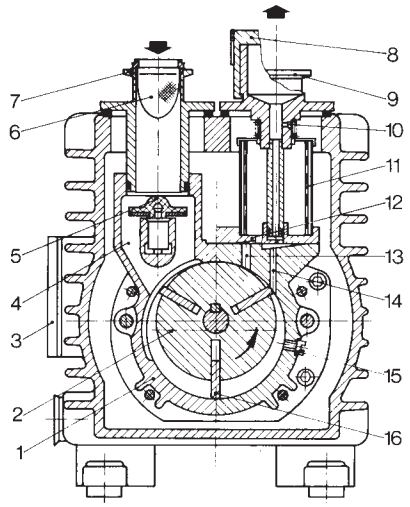
Constant, minimum clearance a for the entire sealing passage b

Fig. 2.4 Arrangement of the sealing passage in rotary vane pumps also known as "duo seal"

As shown in the classification Table 2.1, the oil sealed displacement pumps include rotary vane and rotary plunger pumps of single and two-stage design as well as single-stage trochoid pumps which today are only of historic interest. Such pumps are all equipped with a gas ballast facility which was described in detail (for details see 2.1.2.2.4) for the first time by Gaede (1935). Within specified engineering limits, the gas ballast facility permits pumping of vapors (water vapor in particular) without condensation of the vapors in the pump.

2.1.2.2.1 Rotary vane pumps (TRIVAC A, TRIVAC B, TRIVAC E, SOGEVAC)

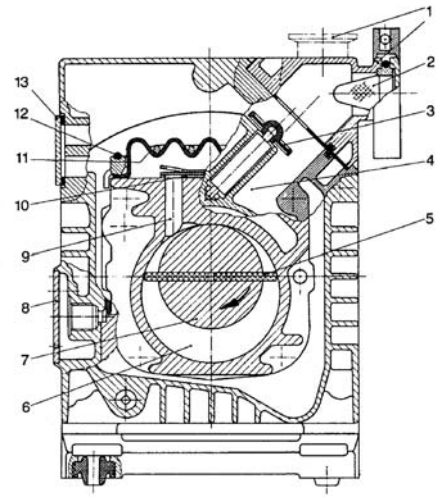
Rotary vane pumps (see also Figs. 2.5 and 2.6) consist of a cylindrical housing (pump-ing ring) (1) in which an eccentrically suspended and slotted rotor (2) turns in the direction of the arrow. The rotor has vanes (16) which are forced outwards usually by centrifugal force but also by springs so that



1 Pump housing
2 Rotor
3 Oil-level sight glass
4 Suction duct
5 Anti-suckback valve
6 Dirt trap
7 Intake port
8 Lid of gas ballast valve
9 Exhaust port
10 Air inlet silencer
11 Oil filter
12 Exhaust valve
13 Exhaust duct
14 Gas ballast duct
15 Oil injection
16 Vane

Fig. 2.5 Cross section of a single-stage rotary vane pump (TRIVAC A)

Vacuum generation



1 Intake port
2 Dirt trap
3 Anti-suckback valve
4 Intake duct
5 Vane
6 Pumping chamber
7 Rotor
8 Orifice, connection for inert gas ballast
9 Exhaust duct
10 Exhaust valve
11 Demister
12 Spring
13 Orifice; connection for oil filter

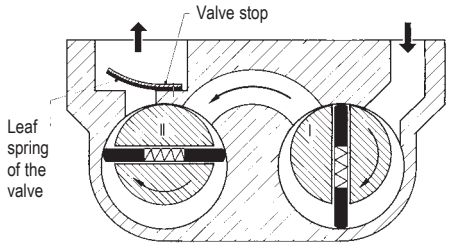
Fig. 2.6 Cross section of a single-stage rotary vane pump (TRIVAC B)

the vanes slide inside the housing. Gas entering through the intake (4) is pushed along by the vanes and is finally ejected from the pump by the oil sealed exhaust valve (12).

The older range of TRIVAC A pumps (Fig. 2.5) from LEYBOLD has three radial vanes offset by 120°. The TRIVAC B range (Fig. 2.6) has only two vanes offset by 180°. In both cases the vanes are forced outwards by the centrifugal forces without the use of springs. At low ambient temperatures this possibly requires the use of a thinner oil. The A-Series is lubricated through the arising pressure difference whereas the B-Series pumps have a geared oil pump for pressure lubrication. The TRIVAC B-Series is equipped with a particularly reliable anti-suckback valve; a horizontal or vertical arrangement for the intake and exhaust ports. The oil level sight glass and the gas ballast actuator are all on the same side of the oil box (user friendly design). In combination with the TRIVAC BCS system it may be equipped with a very comprehensive range of accessories, designed chiefly for semiconductor applications. The oil reservoir of the rotary vane pump and also that of the other oil sealed displacement pumps serves the purpose of lubrication and sealing, and also to fill dead spaces and slots. It removes the heat of gas compression, i.e. for cooling purposes. The oil provides a seal between rotor and pump ring. These parts are “almost” in contact along a straight line (cylinder jacket line). In order to increase the oil sealed surface area a so-called sealing passage is integrated into the pumping ring (see Fig. 2.4). This provides a better seal and allows a higher compression ratio or a lower ultimate pressure. LEYBOLD manufactures three different ranges of rotary vane pumps which are specially adapted to different applications such as high intake pressure, low ultimate pressure or applications in the semiconductor industry. A summary of the more important characteristics of these ranges is given in Table 2.2. The TRIVAC rotary vane pumps are produced as single-stage (TRIVAC S) and two-stage (TRIVAC D) pumps (see Fig. 2.7). With the two-stage oil sealed pumps it is possible to attain lower operating and ultimate pressures

	TRIVAC A	TRIVAC B	TRIVAC BCS	TRIVAC E	SOGEVAC
Vanes per stage	3	2	2	2	3 (tangential)
Pumping speed [m_3/h]	1 – 1.5 2 – 4 8 – 16 30 – 60	1.6 4 – 8 16 – 25 40 – 65	– 16 – 25 40 – 65 –	– 2.5 – –	16 – 25 40 – 100 180 – 280 585 – 1200
Sealing passage	yes	yes	yes	yes	no
Ultimate pressure, single-stage [mbar]	$< 2 \cdot 10^{-2}$	$< 2 \cdot 10^{-2}$	$< 2 \cdot 10^{-2}$	–	$< 5 \cdot 10^{-1}$
Ultimate pressure two-stage [mbar]	$< 2.5 \cdot 10^{-4}$	$< 1 \cdot 10^{-4}$	$< 1 \cdot 10^{-4}$	$< 1 \cdot 10^{-4}$	–
Oil supply	Pressure difference	Gear pump	Gear pump	Eccentric pump	Pressure difference
Slots	Comparable for all types: about 0.01 to 0.05 mm				
Bearing/lubrication	Axial face / oil	Axial face / oil	Axial face / oil	Ball / grease	Ball / oil
Special characteristics	–	Hydropneumatic anti-suckback valve	Coated parts in contact with medium	Many accessories	Cost-effective
Media	No ammonia	Clean to light particles	Aggressive and corrosive	Clean to light particles	Clean
Main areas of application	Multi-purpose	Multi-purpose	Semiconductor industry	Multi-purpose	Packaging industry

Table 2.2 Rotary vacuum pump ranges



I High vacuum stage
II Second forevacuum stage

Fig. 2.7 Cross section of a two-stage rotary vane pump, schematic

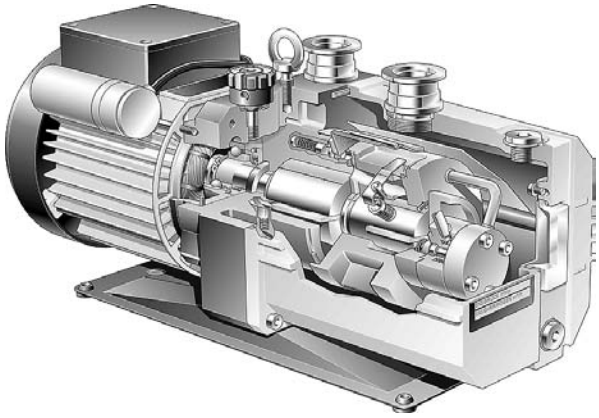


Fig. 2.8a Cross section of a two-stage rotary vane pump (TRIVAC E)

compared to the corresponding single-stage pumps. The reason for this is that in the case of single-stage pumps, oil is unavoidably in contact with the atmosphere outside, from where gas is taken up which partially escapes to the vacuum side thereby restricting the attainable ultimate pressure. In the oil sealed two-stage displacement pumps manufactured by LEYBOLD, oil which has already been degassed is supplied to the stage on the side of the vacuum (stage 1 in Fig. 2.7): the ultimate pressure lies almost in the high vacuum range, the lowest operating pressures lie in the range between medium vacuum / high vacuum. Note: operating the so called high vacuum stage (stage 1) with only very little oil or no oil at all will – in spite of the very low ultimate pressure – in practice lead to considerable difficulties and will significantly impair operation of the pump.

2.1.2.2.2 Rotary plunger pumps (E Pumps)

Shown in Fig. 2.9 is a sectional view of a rotary plunger pump of the single block type. Here a piston (2) which is moved along by an eccentric (3) turning in the direction of the arrow moves along the chamber wall. The gas which is to be pumped flows into the pump through the intake port (11), passes through the intake channel of the slide valve (12) into the pumping chamber (14). The slide valve forms a unit with the piston and slides to and fro between the rotatable valve guide in the casing (hinge bar 13). The gas drawn into the pump finally enters the compression chamber (4). While turning, the piston compresses this quantity of gas until it is ejected through the oil sealed valve (5). As in the case of rotary vane pumps, the oil reservoir is used for lubrication, sealing, filling of dead spaces and cooling. Since the pumping chamber is divided by the piston into two spaces, each turn completes an operating cycle (see Fig. 2.10). Rotary plunger pumps are manufactured as single and two-stage pumps. In many vacuum processes combining a Roots pump with a single-stage rotary plunger pump may offer

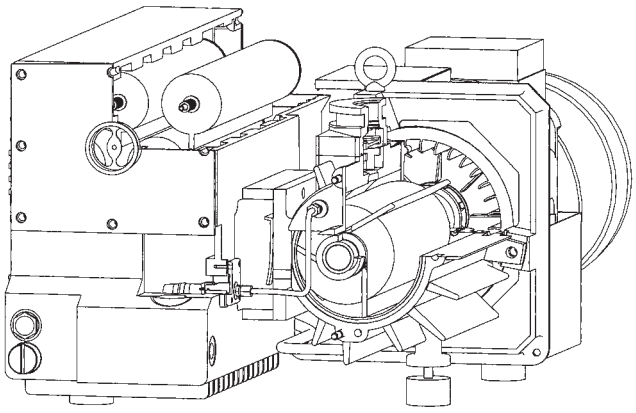
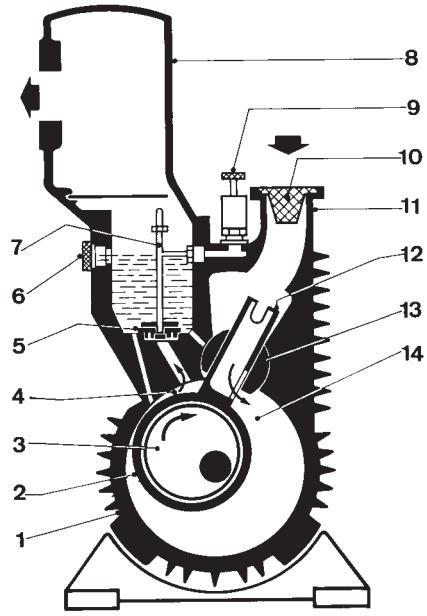


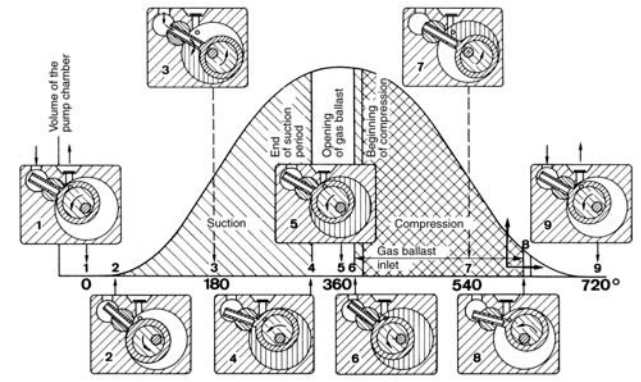
Fig. 2.8b SOGEVAC pump SV 300 with three tangential vanes



1 Casing
2 Cylindrical piston
3 Eccentric
4 Compression chamber
5 Oil sealed pressure valve
6 Oil-level sight glass
7 Gas ballast channel
8 Exhaust pot
9 Gas ballast valve
10 Dirt trap
11 Intake port
12 Slide valve
13 Hinge bar
14 Pumping chamber (air is flowing in).

Fig. 2.9 Cross section of a single-stage rotary plunger pump (monoblock design)

Vacuum generation



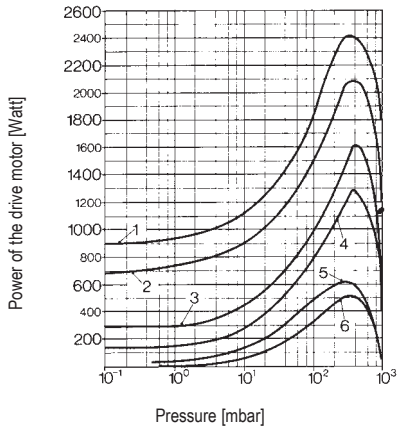
1 Upper dead point
2 Slot in suction channel of slide valve is freed – beginning of suction period
3 Lower dead point – slot in suction channel is quite free, and pumped-in gas (arrow) enters freely into the pumping chamber (shown shaded)
4 Slot in suction channel is closed again by swivelling hinge bar – end of suction period
5 Upper dead point – maximum space between rotating piston and stator
6 Shortly before beginning of compression period, the front surface of the rotating plunger frees gas ballast opening – commencement of gas ballast inlet
7 Gas ballast opening is quite free
8 End of gas ballast inlet
9 End of pumping period.

Fig. 2.10 Operating cycle of a rotary plunger pump (for positions 1 to 9 of the plunger)

more advantages than a two-stage rotary plunger pump alone. If such a combination or a two-stage pump is inadequate, the use of a Roots pump in connection with a two-stage pump is recommended. This does not apply to combinations involving rotary vane pumps and Roots pumps.

Motor power

The motors supplied with the rotary vane and rotary plunger pumps deliver enough power at ambient temperatures of 12 °C and when using our special oils to cover the maximum power requirement (at about 400 mbar). Within the actual operating range of the pump, the drive system of the warmed up pump needs to supply only about one third of the installed motor power (see Fig. 2.11).



1 Operating temp. curve 1 32°C,
2 Operating temp. curve 2 40°C,
3 Operating temp. curve 3 60°C,
4 Operating temp. curve 4 90°C,
5 Theoretic curve for adiabatic compression
6 Theoretic curve for isotherm compression

Fig. 2.11 Motor power of a rotary plunger pump (pumping speed 60 m³/h) as a function of intake pressure and operating temperature. The curves for gas ballast pumps of other sizes are similar.

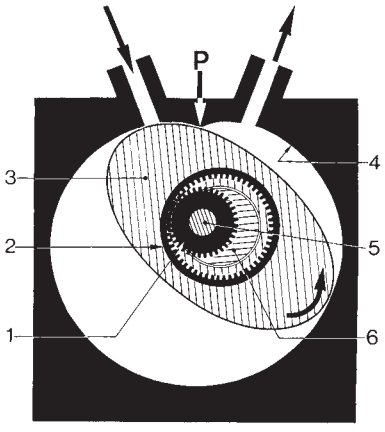
2.1.2.2.3 Trochoid pumps

Trochoid pumps belong to the class of so called rotary piston pumps, which (see overview of Table 2.1) in turn belong to the group of rotary pumps. With rotary piston pumps, the piston's center of gravity runs along a circular path about the rotational axis (hence rotary piston machines). A rotary piston pump can – in contrast to the rotary plunger pump – be completely balanced dynamically. This offers the advantage that larger pumps can operate without vibration so that they can be installed without needing foundations. Moreover, such pumps may be operated at higher speed, compared to rotary plunger pumps (see below). The volume of the pumping chamber with respect to the volume of the entire pump – the so called specific volume – is, in the case of trochoid pumps, approximately twice of that of rotary plunger pumps. Larger rotary plunger pumps run at speeds of 500 rpm. The trochoid pump may run at 1000 rpm and this applies also to larger designs. It is thus about four times smaller compared to a rotary plunger pump having the same pumping speed and runs without producing any vibrations. Unfortunately the advantages in the area of engineering are combined with great disadvantages in the area of manufacturing, so that today LEYBOLD does not produce trochoid pumps any more. Operation of such a pump is shown in the sectional diagram of Fig. 2.12.

2.1.2.2.4 The gas ballast

The gas ballast facility as used in the rotary vane, rotary plunger and trochoid pumps permits not only pumping of permanent gases but also even larger quantities of condensable gases.

The gas ballast facility (see Fig. 2.13) prevents condensation of vapors in the pump chamber of the pump. When pumping vapors these may only be compressed up to their saturation vapor pressure at the temperature of the pump. If pumping water vapor, for example, at a pump temperature of 70 °C, the vapor may only be compressed to 312 mbar (saturation vapor pressure of water at 70 °C (see Table XIII in Section 9)). When compressing further, the water vapor condenses without increasing the



1 Toothed wheel fixed to the driving shaft
2 Toothed wheel fixed to the piston
3 Elliptic piston
4 Inner surface of the pump
5 Driving shaft
6 Eccentric

Fig. 2.12 Cross section of a trochoid pump

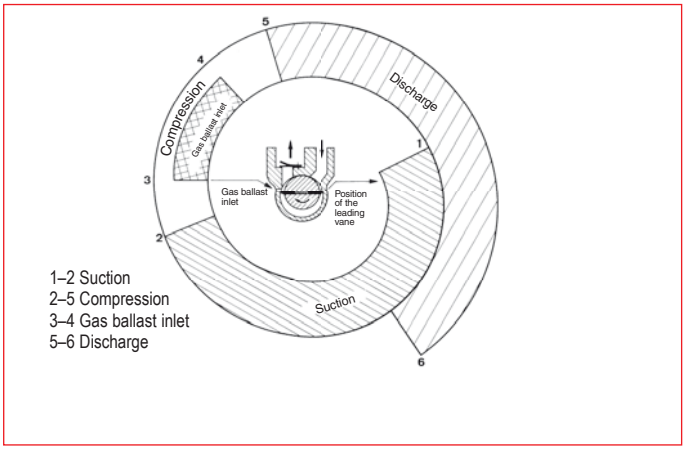


Fig. 2.13 Working process within a rotary vane pump with gas ballast

pressure. No overpressure is created in the pump and the exhaust valve is not opened. Instead the water vapor remains as water in the pump and emulsifies with the pump's oil. This very rapidly impairs the lubricating properties of the oil and the pump may even seize when it has taken up too much water. The gas ballast facility developed in 1935 by Wolfgang Gaede inhibits the occurrence of condensation of the vapor in the pump as follows. Before the actual compression process begins (see Fig. 2.13), a precisely defined quantity of air ("the gas ballast") is admitted into the pumping chamber of the pump. The quantity is such that the compression ratio of the pump is reduced to 10:1 max. Now vapors which have been taken in by the pump may be compressed together with the gas ballast, before reaching their condensation point and ejected from the pump. The partial pressure of the vapors which are taken in may however not exceed a certain value. It must be so low that in the case of a compression by a factor of 10, the vapors can not condense at the operating temperature of the pump. When pumping water vapor this critical value is termed the "water vapor tolerance".

Shown schematically in Fig. 2.14 is the pumping process with and without gas ballast as it takes place in a rotary vane pump when pumping condensable vapors.

Two requirements must be met when pumping vapors:

1) the pump must be at operating temperature.

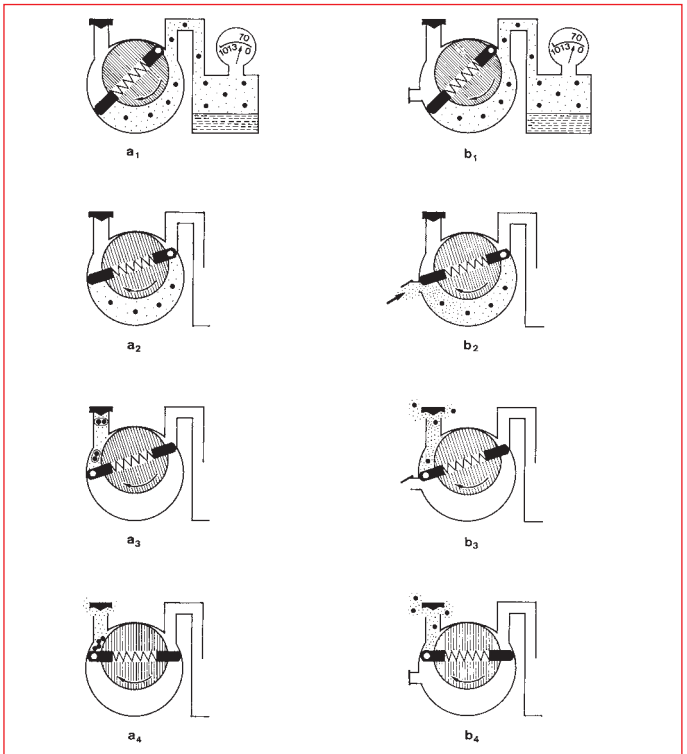
2) the gas ballast valve must be open.

(With the gas ballast valve open the temperature of the pump increases by about 10 °C. Before pumping vapors the pump should be operated for half an hour with the gas ballast valve open).

Simultaneous pumping of gases and vapors

When simultaneously pumping permanent gases and condensable vapors from a vacuum system, the quantity of permanent gas will often suffice to prevent any condensation of the vapors inside the pump. The quantity of vapor which may be pumped without condensation in the pump can be calculated as follows:

$$\frac{p_{vapour}}{p_{vapour} + p_{perm}} < \frac{p_{vapour,\,sat}}{p_{sum}} \qquad (2.1)$$



a) Without gas ballast
1) Pump is connected to the vessel, which is already almost empty of air (70 mbar) – it must thus transport mostly vapor particles
2) Pump chamber is separated from the vessel – compression begins
3) Content of pump chamber is already so far compressed that the vapor condenses to form droplets – overpressure is not yet reached
4) Residual air only now produces the required overpressure and opens the discharge valve, but the vapor has already condensed and the droplets are precipitated in the pump.

b) With gas ballast
1) Pump is connected to the vessel, which is already almost empty of air (70 mbar) – it must thus transport mostly vapor particles
2) Pump chamber is separated from the vessel – now the gas ballast valve, through which the pump chamber is filled with additional air from outside, opens – this additional air is called gas ballast
3) Discharge valve is pressed open, and particles of vapor and gas are pushed out – the overpressure required for this to occur is reached very early because of the supplementary gas ballast air, as at the beginning the entire pumping process condensation cannot occur
4) The pump discharges further air and vapor

Fig. 2.14 Diagram of pumping process in a rotary vane pump without (left) and with (right) gas ballast device when pumping condensable substances.

Where:

p_{vapor} = is the partial pressure of vapor at the intake of the pump

p_{perm} = is the total pressure of all pumped permanent gases at the intake of the pump

$p_{vapor,sat}$ = is the saturation pressure of the pumped vapor, depending on temperature (see Fig. 2.15)

p_{sum} = $p_{exhaust} + \Delta p_{valve} + \Delta p_{exhaust\ filter}$

Δp_{valve} = is the pressure difference across the exhaust valve which amounts depending on type of pump and operating conditions to 0.2 ... 0.4 bar

$\Delta p_{exhaust\ filter}$ = is the pressure difference across the exhaust filter amounting to 0 ... 0.5 bar

Vacuum generation

Example 1:
With a rotary vane pump with an external oil mist filter in series, a mixture of water vapor and air is being pumped. The following values are used for applying eq. (2.1):

$p_{exhaust}$ = 1 bar

$\Delta p_{valve} + \Delta p_{exhaust\ filter}$ = 0.35 bar,
temperature of the pump 70 °C
Hence:
p_{sum} = 1.35 bar; $p_{vapor\ sat}$ (H_2O)
= 312 mbar
(see Table XIII in chapter 9)

Using eq. (2.1) follows:

$$\frac{p_{vapour,\ H_2O}}{p_{vapour,\ H_2O} + p_{air}} < \frac{312}{1350} = 0.23$$

The pressure of the water vapor in the air/water vapor mixture must not exceed 23 % of the total pressure of the mixture.

Example 2:
Ethanoic acid is to be pumped with a rotary plunger pump.

$p_{exhaust}$ = 1.1 bar (taking into consideration the flow resistance of the pipes)
Δp_{valve} = 0.25 bar
$\Delta p_{exhaust\ filter}$ = 0.15 bar
(pressure loss in the oil mist trap)

Hence:
p_{sum} = 1.5 bar.

By controlled cooling the pump and oil temperature is set at 100 °C. The saturation pressure of the acid therefore is – see Fig. 2.15 – $p_{vapor,\ sat}$ = 500 mbar.

From eq. (2.1) follows:

$$\frac{p_{vapour,\ acid}}{p_{vapour,\ acid} + p_{air}} = < \frac{0.5}{1.5} = \frac{1}{3}$$

Returning to the question of pumping water vapor in a mixture with air, the ratio 3 parts of permanent gases to 1 part of water vapor, as indicated in example 1, can be for guidance only. In actual practice it is recommended to run up a rotary pump of the types described hitherto always with the gas ballast valve open, because it takes some time until the pump has reached its final working temperature.

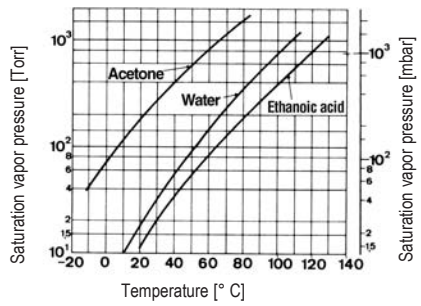


Fig. 2.15 Saturation vapor pressures

From eq. (2.1) follows for the permissible partial pressure p_{vapor} of the pumped vapor the relation

$$p_{vapour} \leq \frac{p_{vapour,\ sat}}{p_{sum} - p_{vapour,\ sat}} \cdot p_{perm} \qquad (2.2)$$

This relation shows that with p_{perm} = 0 no vapors can be pumped without condensation in the pump, unless the gas-ballast concept is applied. The corresponding formula is:

$$p_{vapour} \leq \frac{B}{S} p_{sum} \cdot \frac{(p_{Vapour,\ sat} - p_{vapour,\ g.b.})}{p_{sum} - p_{vapour,\ sat}} + \frac{p_{vapour\ sat}}{p_{sum} - p_{vapour\ sat}} p_{perm} \qquad (2.3)$$

Where:

B = is the volume of air at 1013 mbarwhich is admitted to the pump chamber per unit time, called in brief the “gas ballast”

S = is the nominal speed of the pump (volume flow rate)

p_{sum} = is the pressure at the discharge outlet of the pump, assumed to be a maximum at 1330 mbar

$p_{vapor,\ sat}$ = Saturation vapor pressure of the vapor at the pump’s exhaust port

$p_{vapor,\ g.b.}$ = is the partial pressure of any vapor that might be present in the gas used as gas ballast (e.g. water vapor contained in the atmospheric air when used as gas ballast)

p_{perm} = is the total pressure of all permanent gases at the inlet port of the pump

Eq. (2.3) shows that when using gas ballast (B 0) vapors can also be pumped without condensation if no gas is present at the intake of the pump. The gas ballast may also be a mixture of non-condensable gas and condensable vapor as long as the partial pressure of this vapor ($p_{vapor,\ g.b.}$) is less than the saturation pressure $p_{vapor',\ sat}$ of the pumped vapor at the temperature of the pump.

Water vapor tolerance
An important special case in the general considerations made above relating to the topic of vapor tolerance is that of pumping water vapor. According to PNEUROP water vapor tolerance is defined as follows:

“Water vapor tolerance is the highest pressure at which a vacuum pump, under normal ambient temperatures and pressure conditions (20 °C, 1013 mbar), can continuously take in and transport **pure** water vapor. It is quoted in mbar”. It is designated as $p_{W,O}$.

Applying eq. (2.3) to this special case means:

p_{perm} = 0 and $p_{vapor,\ sat} = p_s$ (H_2O), thus:

$$p_{W,0} = \frac{B}{S}\, p_{sum}\, \frac{p_s(H_2O) - p_{vapour,\ g.b.}}{p_{sum} - p_s(H_2O)} \qquad (2.4)$$

If for the gas ballast gas atmospheric air of 50 % humidity is used, then $p_{vapor,\ g.b.}$ = 13 mbar; with B/S = 0.10 – a usual figure in practice – and p_{sum} (total exhaust pressure) = 1330 mbar, the water vapor tolerance $p_{W,0}$ as function of the temperature of the pump is represented by the lowest curve in diagram Fig. 2.16. The other curves correspond to the pumping of water vapor-air mixtures, hence $p_{perm} = p_{air}$ O), indicated by the symbol p_L in millibar. In these cases a higher amount of water vapor partial pressure p_w can

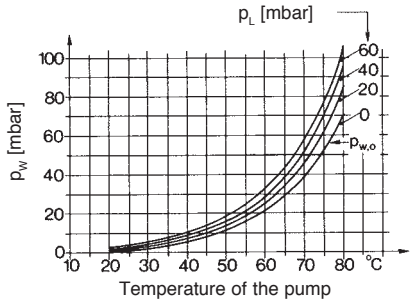


Fig. 2.16 Partial pressure p_w of water vapor that can be pumped with the gas ballast valve open without condensation in the pump, as a function of the pump temperature for various partial pressures p_L of air. The lowest curve corresponds to the water vapor tolerance $p_{w,o}$ of the pump.

be pumped as shown in the diagram. The figures for $p_{W,0}$ given in the catalogue therefore refer to the lower limit and are on the safe side.

According to equation 2.4 an increase in the gas ballast B would result in an increased water vapor tolerance $p_{W,0}$. In practice, an increase in B, especially in the case of single-stage gas ballast pumps is restricted by the fact that the attainable ultimate vacuum for a gas ballast pump operated with the gas ballast valve open becomes worse as the gas ballast B increases. Similar considerations also apply to the general equation 2.3 for the vapor tolerance p_{vapor}.

At the beginning of a pump down process, the gas ballast pump should always be operated with the gas ballast valve open. In almost all cases a thin layer of water will be present on the wall of a vessel, which only evaporates gradually. In order to attain low ultimate pressures the gas ballast valve should only be closed after the vapor has been pumped out. LEYBOLD pumps generally offer a water vapor tolerance of between 33 and 66 mbar. Two-stage pumps may offer other levels of water vapor tolerance corresponding to the compression ratio between their stages – provided they have pumping chamber of different sizes.

Other gases as ballast

Generally atmospheric air is used as the gas ballast medium. In special cases, when pumping explosive or toxic gases, for example, other permanent gases like noble gases or nitrogen, may be used (see Section 8.3.1.3).

2.1.3 Dry compressing rotary displacement pumps

2.1.3.1 Roots pumps

The design principle of the Roots pumps was already invented in 1848 by Isaiah Davies, but it was 20 years later before it was implemented in practice by the Americans Francis and Philander Roots. Initially such pumps were used as blowers for combustion motors. Later, by inverting the drive arrangement, the principle was employed in gas meters. Only since 1954 has this principle been employed in vacuum engineering. Roots pumps are used in pump combinations together with backing pumps (rotary vane- and rotary plunger pumps) and extend their operating range well into the medium vacuum range. With two stage Roots pumps this extends into the high vacuum range. The operating principle of Roots pumps permits the assembly of units having very high pumping speeds (over 100,000 m^3/h) which often are more economical to operate than steam ejector pumps running in the same operating range.

A Roots vacuum pump (see Fig. 2.17) is a rotary positive-displacement

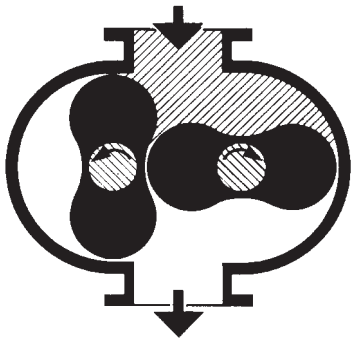


Fig. 2.17 Schematic cross section of a Roots pump

type of pump where two symmetrically-shaped impellers rotate inside the pump casing past each other in close proximity. The two rotors have a cross section resembling approximately the shape of a figure 8 and are synchronized by a toothed gear. The clearance between the rotors and the casing wall as well as between the rotors themselves amounts only to a few tenths of a millimeter. For this reason Roots pumps may be operated at high speeds without mechanical wear. In contrast to rotary vane and rotary plunger pumps, Roots pumps are not oil sealed, so that the internal leakage of dry compressing pumps by design results in the fact that compression ratios only in the range 10 – 100 can be attained. The internal leakage of Roots pumps, and also other dry compressing pumps for that matter, is mainly based on the fact that owing to the operating principle certain surface areas of the pump chamber are assigned to the intake side and the compression side of the pump in alternating fashion. During the compression phase these surface areas (rotors and casing) are loaded with gas (boundary layer); during the suction phase this gas is released. The thickness of the traveling gas layer depends on the clearance between the two rotors and between the rotors and the casing wall. Due to the relatively complex thermal conditions within the Roots pump it is not possible to base one's consideration on the cold state. The smallest clearances and thus the lowest back flows are attained at operating pressures in the region of

1 mbar. Subsequently it is possible to attain in this region the highest compression ratios, but this pressure range is also most critical in view of contacts between the rotors and the casing.

Characteristic quantities of roots pumps

The quantity of gas Q_{eff} effectively pumped by a Roots pump is calculated from the theoretically pumped quantity of gas Qth and the internal leakage QiR (as the quantity of gas which is lost) as:

$$Q_{eff} = Q_{th} - Q_{iR} \quad (2.5)$$

The following applies to the theoretically pumped quantity of gas:

$$Q_{th} = p_a \cdot S_{th} \quad (2.6)$$

where pa is the intake pressure and S_{th} is the theoretical pumping speed. This in turn is the product of the pumping volume V_S and the speed n:

$$S_{th} = n \cdot V_S \quad (2.7)$$

Similarly the internal leakage Q_{iR} is calculated as:

$$Q_{iR} = n \cdot V_{iR} \quad (2.8)$$

where p_V is the forevacuum pressure (pressure on the forevacuum side) and S_{iR} is a (notional) "reflow" pumping speed with

$$S_{iR} = n \cdot V_{iR} \quad (2.9)$$

i.e. the product of speed n and internal leakage volume V_{iR}.

Volumetric efficiency of a Roots pumps is given by

$$\eta = \frac{Q_{eff}}{Q_{th}} \quad (2.10)$$

By using equations 2.5, 2.6, 2.7 and 2.8 one obtains

$$\eta = 1 - \frac{p_V}{p_a} \cdot \frac{S_{iR}}{S_{th}} \quad (2.11)$$

When designating the compression p_V/p_a as k one obtains

$$\eta = 1 - k \frac{S_{iR}}{S_{th}} \quad (2.11a)$$

Maximum compression is attained at zero throughput (see PNEUROP and DIN 28 426, Part 2). It is designated as k_0:

$$k_0 = \left(\frac{S_{th}}{S_{iR}}\right)_\eta = 0 \quad (2.12)$$

k_0 is a characteristic quantity for the Roots pump which usually is stated as a function of the forevacuum pressure p_V (see Fig. 2.18). k_0 also depends (slightly) on the type of gas.

For the efficiency of the Roots pump, the generally valid equation applies:

$$\eta = 1 - \frac{k}{k_0} \quad (2.13)$$

Normally a Roots pump will be operated in connection with a downstream rough vacuum pump having a nominal pumping speed S_V. The continuity

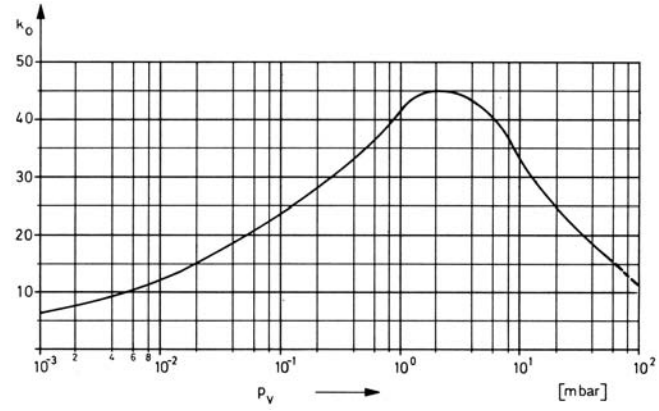


Fig. 2.18 Maximum compression k_0 of the Roots pump RUVAC WA 2001 as a function of fore vacuum pressure p_V

equation gives:

$$S_V \cdot p_V = S_{eff} \cdot p_a = \eta \cdot S_{th} \cdot p_a \quad (2.14)$$

From this

$$k = \frac{p_V}{p_a} = \eta \cdot \frac{S_{th}}{S_V} \quad (2.15)$$

The ratio S_{th}/S_V (theoretical pumping speed of the Roots pump / pumping speed of the backing pump) is termed the gradation k_{th}. From (2.15) one obtains

$$k = \eta \cdot k_{th} \quad (2.16)$$

Equation (2.16) implies that the compression k attainable with a Roots pump must always be less than the grading k_{th} between Roots pump and backing pump since volumetric efficiency is always < 1. When combining equations (2.13) and (2.16) one obtains for the efficiency the well known expression

$$\eta = \frac{k_0}{k_0 + k_{th}} \quad (2.17)$$

The characteristic quantities to be found in equation 2.17 are only for the combination of the Roots pump and the backing pump, namely maximum compression k_0 of the Roots pump and gradation k_{th} between Roots pump and backing pump.

With the aid of the above equations the pumping speed curve of a given combination of Roots pump and backing pump may be calculated. For this the following must be known:

a) the theoretical pumping speed of the Roots pump: S_{th}

b) the max. compression as a function of the fore vacuum pressure: k_0 (p_V)

c) the pumping speed characteristic of the backing pump S_V (p_V)

The way in which the calculation is carried out can be seen in Table 2.3 giving the data for the combination of a Roots pump **RUVAC WA 2001 / E 250** (single-stage rotary plunger pump, operated without gas ballast).

In this the following is taken for S_{th}:

$$S_{th} = 2050 - 2.5\ \% = 2000\ m^3/h$$

The method outlined above may also be applied to arrangements which consist of a rotary pump as the backing pump and several Roots pumps connected in series, for example. Initially one determines – in line with an iteration method – the pumping characteristic of the backing pump plus the first Roots pump and then considers this combination as the backing pump for the second Roots pump and so on. Of course it is required that the theoretical pumping speed of all pumps of the arrangement be known and that the compression at zero throughput k_0 as a function of the backing pressure is also known. As already stated, it depends on the vacuum process which grading will be most suitable. It may be an advantage when backing pump and Roots pump both have the same pumping speed in the rough vacuum range.

Power requirement of a roots pump

Compression in a Roots pump is performed by way of external compression and is termed as isochoric compression. Experience shows that the following equation holds approximately:

$$N_{compression} = S_{th} (p_v - p_a) \qquad (2.18)$$

In order to determine the total power (so-called shaft output) of the pump, mechanical power losses N_V (for example in the bearing seals) must be considered:

$$N_{tot} = N_{compression} + \sum N_V \qquad (2.19)$$

The power losses summarized in N_V are – as shown by experience – approximately proportional to S_{th}, i.e.:

$$\sum N_v = \text{const} \cdot S_{th} \qquad (2.20)$$

Depending on the type of pump and its design the value of the constant ranges between 0.5 and 2 Wh / m^3 .

The total power is thus:

$$N_{tot} = S_{th} (p_v - p_a + \text{const.})$$

The corresponding numerical value equation which is useful for calculations is:

$$N_{tot} = S_{th} (p_v - p_a + \text{const.}) \cdot 3 \cdot 10^{-2}\ \text{Watt} \qquad (2.21)$$

with p_v, p_a in mbar, S_{th} in m^3 / h and the constant “const.” being between 18 and 72 mbar.

Forevacuum pressure P_v	Pumping speed S_v of the E 250	$k_{th} = S_{th} / S_v = 2001/S_v$	$k_0 (p_v)$ of the RUVAC WA 2001	$\eta = k_0 / k_0 + k_{th}$ (Volumetric. efficiency)	$S_{eff} = \eta\ S_{th}$ (equation 2.14)	Intake pressure $p_a = p_v \cdot S_v / S_{eff}$
100	250	8.0	12.5	0.61	1.220	21
40	250	8.0	18	0.69	1.380	7.2
10	250	8.0	33	0.8	1.600	1.6
5	250	8.0	42	0.84	1.680	0.75
1	250	8.0	41	0.84	1.680	0.15
$5 \cdot 10^{-1}$	220	9.1	35	0.79	1.580	$7 \cdot 10^{-2}$
$1 \cdot 10^{-1}$	120	16.6	23	0.6	1.200	$1 \cdot 10^{-2}$
$4 \cdot 10^{-2}$	30	67	18	0.21	420	$3 \cdot 10^{-3}$
The values taken from the two right-hand columns give point by point the pumping speed curve for the combination WA 2001/E250 (see Fig. 2.19, topmost curve)					↓ Pumping speed characteristic for the combination WA 2001 / E250	↓

Table 2.3

Vacuum generation

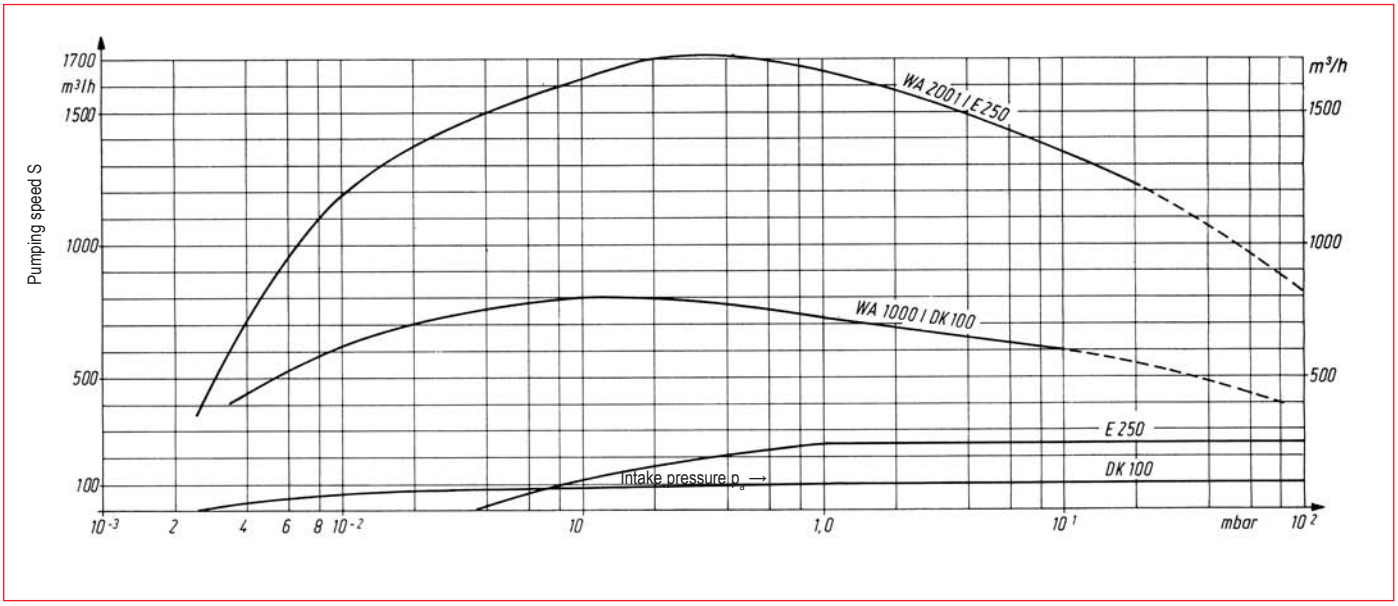


Fig. 2.19 Pumping speed curves for different pump combinations with the corresponding backing pumps

Load rating of a roots pump

The amount of power drawn by the pump determines its temperature. If the temperature increases over a certain level, determined by the maximum permissible pressure difference $p_V - p_a$, the danger exists that the rotors may seize in the casing due to their thermal expansion. The maximum permissible pressure difference Δp_{max} is influenced by the following factors: forevacuum or compression pressure p_V, pumping speed of the backing pump S_V, speed of the Roots pump n, gradation k_{th} and the adiabatic exponent κ of the pumped gas. Δp_{max} increases when p_V and S_V increase and decreases when n and k_{th} increase. Thus the maximum difference between forevacuum pressure and intake pressure, $p_V - p_a$ must – during continuous operation – not exceed a certain value depending on the type of pump. Such values are in the range between 130 and 50 mbar. However, the maximum permissible pressure difference for continuous operation may be exceed for brief periods. In the case of special designs, which use gas cooling, for example, high pressure differences are also permissible during continuous operation.

Types of motors used with roots pumps

Standard flange-mounted motors are used as the drive. The shaft feedthroughs are sealed by two oil sealed radial shaft seals running on a wear resistant bushing in order to protect the drive shaft. Flange motors of any protection class, voltage or frequency may be used.

Integral leak tightness of this version is $< 10^{-4}$ mbar · l · s^{-1}.

In the case of better leak tightness requirements of $< 10^{-5}$ mbar · l · s^{-1} the Roots pump is equipped with a canned motor. The rotor is seated in the vacuum on the drive shaft of the pump and is separated from the stator by a vacuum-tight non-magnetic tube. The stator coils are cooled by a fan having its own drive motor. Thus shaft seals which might be subject to wear are no longer required. The use of Roots pumps equipped with canned motors is especially recommended when pumping high purity-, toxic- or radioactive gases and vapors.

Maintaining the allowed pressure difference

In the case of standard Roots pumps, measures must be introduced to ensure that the maximum permissible pressure difference between intake and exhaust port due to design constraints is not exceeded. This is done either by a pressure switch, which cuts the Roots pump in and out depending on the intake pressure, or by using a pressure difference or overflow valve in the bypass of the Roots pumps (Fig. 2.20 and 2.21). The use of an overflow valve in the bypass of the Roots pump is the better and more reliable solution. The weight and spring loaded valve is set to the maximum permissible pressure difference of the particular pump. This ensures that the Roots pump is not overloaded and that it may be operated in any pressure range. In practice this means that the Roots pump can be switched on, together with the backing pump, at atmospheric pressure. In the process any pressure increases will not adversely affect combined operation, i.e. the Roots pump is not switched off in such circumstances.

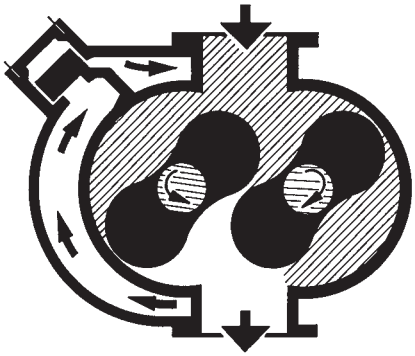


Fig. 2.20 Cross section of a Roots pumps with bypass line

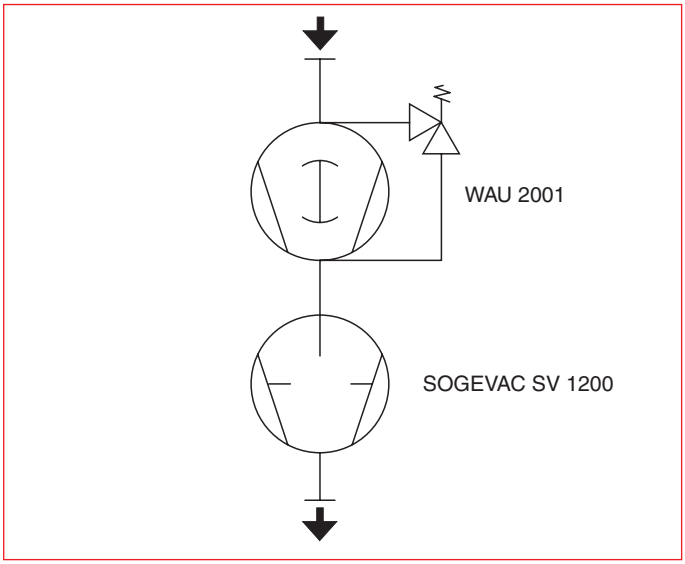


Fig. 2.21 Vacuum diagram – Roots pump with integrated bypass line and backing pump

Pre-admission cooling (Fig. 2.22)
In the case of Roots pumps with pre-admission cooling, the compression process basically is the same as that of a normal Roots pump. Since greater pressure differences are allowed more installed power is needed, which at the given speed and the pressure difference between inlet and discharge port is directly proportional and is composed of the theoretical work done on compression and various power losses. The compression process ends normally after opening of the pumping chamber in the direction of the discharge port. At this moment warmed gas at higher pressure flows into the pumping chamber and compresses the transported volume of gas. This compression process is performed in advance in the case of pre-admission cooling. Before the rotor opens the pumping chamber in the direction of the discharge port, compressed and cooled gas flows into the pumping chamber via the pre-admission channel. Finally the rotors eject the pumped medium via the discharge port. The cooled gas, which in the case of single-stage compression is taken from the atmosphere and admitted from the pre-admission cooler, and which in the case of multi-stage pump systems is taken from downstream gas coolers, performs a pre-compression and removes by "inner cooling" the heat of compression at the point of time it occurs.

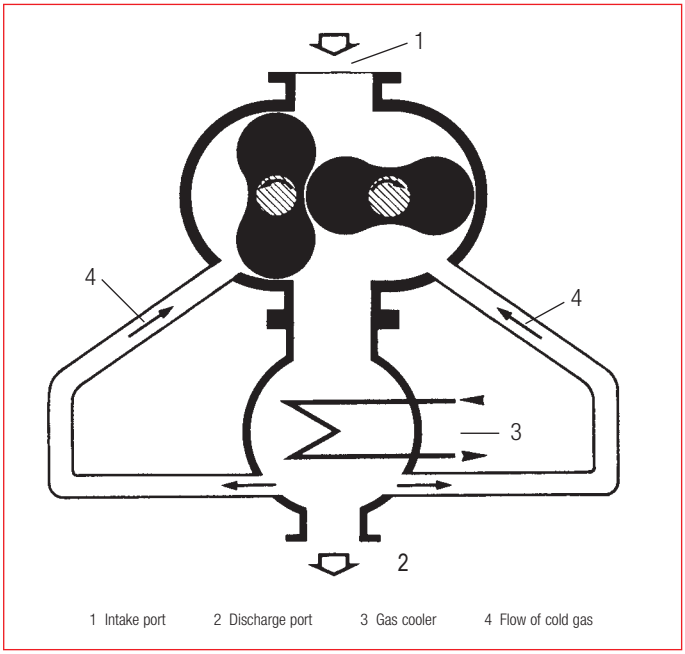


Fig. 2.22 Diagram of a Roots pump with pre-admission cooling

2.1.3.2 Claw pumps

Like Roots pumps, claw pumps belong to the group of dry compressing rotary piston vacuum pumps (or rotary vacuum pumps). These pumps may have several stages; their rotors have the shape of claws.

The **design principle** of a claw pump is explained by first using an example of a four-stage design. The cross section inside the pump's casing has the shape of two partly overlapping cylinders (Fig. 2.23). Within these cylinders there are two freely rotating rotors in each pump stage: (1) with their claws and the matching recesses rotating in opposing directions about their vertical axes. The rotors are synchronized by a gear just like a Roots pump. In order to attain an optimum seal, the clearance between the rotor at the center of the casing and the amount of clearance with respect to the inside casing wall is very small; both are in the order of magnitude of a few 0.01 mm. The rotors periodically open and close the intake and discharge slots (5) and (4). At the beginning of the work cycle in position a, the right rotor just opens the intake slot (5). Gas now flows into the continually increasing intake space (3) in position b until the right rotor seals off the intake slot (5) in position c. After both claws have passed through the center position, the gas which has entered is then compressed in the

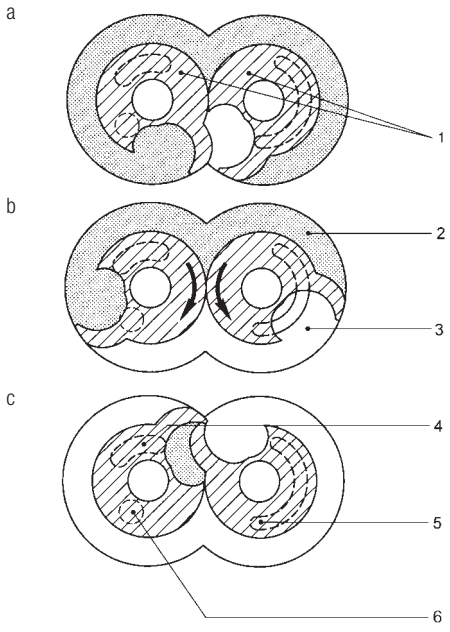


Fig. 2.23 Principle of operation

Vacuum generation

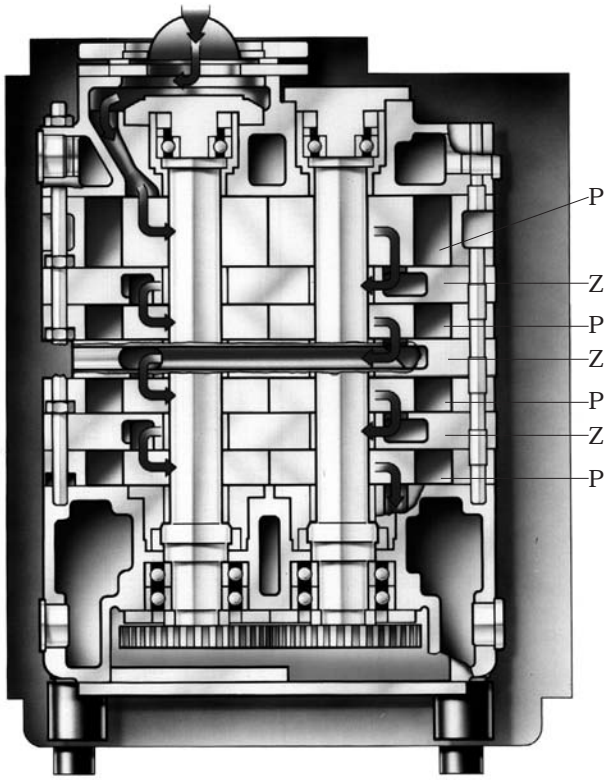


Fig. 2.24 Arrangement of the pumps and guiding of the gas flow. P = Pump stage Z = Intermediate ring

compression chamber (2) (position a) so long until the left rotor releases the discharge slot (4) (position b) thereby discharging the gas. Immediately after the compression process has started (position a) the intake slot (5) is opened simultaneously and gas again flows into the forming intake space (3) (position b). Influx and discharge of the gas is performed during two half periods. Each rotor turns twice during a full work cycle. Located between the pumping stages are intermediate discs with flow channels which run from the discharge side of the upper stage to the intake side of the next stage, so that all inlet or exhaust sides are arranged vertically above each other (Fig. 2.24). Whereas in a Roots pump the incoming gas is pumped through the pump at a constant volume and compression is only performed

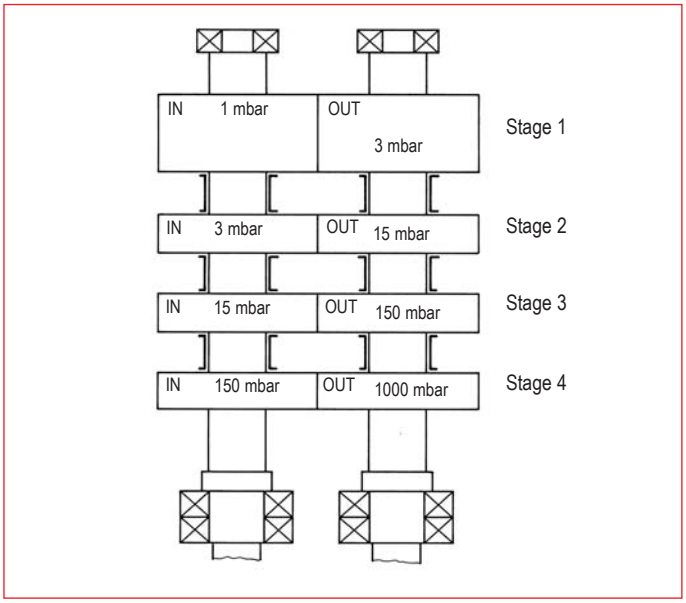


Fig. 2.25 Pressures in pump stages 1 to 4

in the forevacuum line (see Section 2.1.3.1), the claw pump compresses the gas already within the pumping chamber until the rotor releases the discharge slot. Shown in Fig. 2.25 are the average pressure conditions in the individual pumping stages of a DRYVAC at an intake pressure of 1 mbar. In order to meet widely differing requirements LEYBOLD manufactures two different series of claw pumps, which chiefly differ in the type of compression process used:

1) Pumps **with internal compression**, multi-stage for the **semiconductor industry (DRYVAC Series)** and
2) Pumps **without internal compression**, two-stage for **chemistry** applications **("ALL-ex")**.

Figs. 2.26 and 2.27 demonstrate the differences in design. Shown is the course of the pressure as a function of the volume of the pumping chamber by way of a pV diagram.

Fig. 2.26 shows the (polytropic) course of the compression for pumps with internal compression. The pressure increases until the discharge slot is

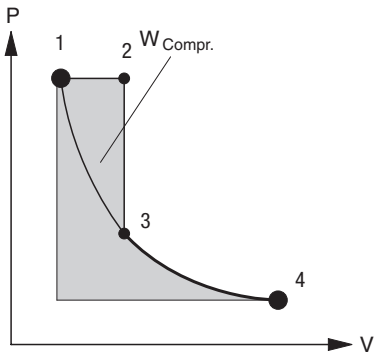


Fig. 2.26 Compression curve for a claw pump with internal compression

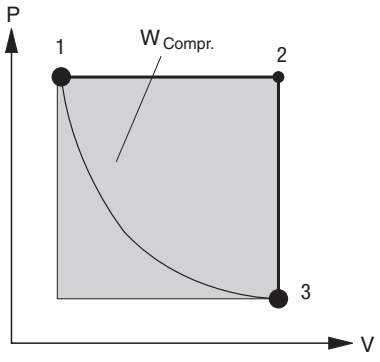
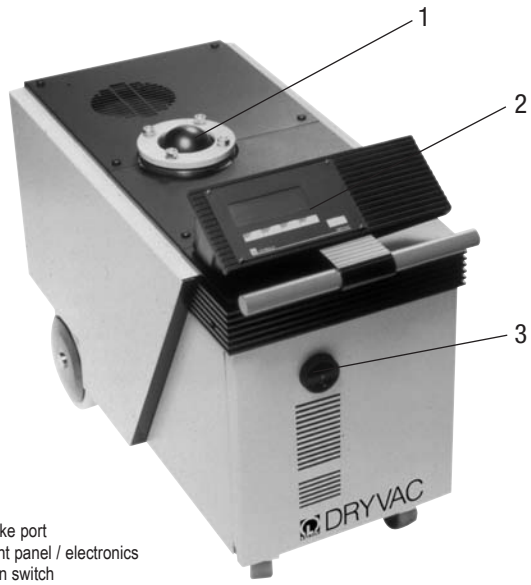


Fig. 2.27 Compression curve for a claw pump without internal compression ("isochoric compression")



1 Intake port
2 Front panel / electronics
3 Main switch

Fig. 2.28 DRYVAC pump

opened. If at that point the exhaust back pressure has not been reached, the compression space is suddenly vented with hot exhaust gas. As the volume is reduced further, the gas at exhaust pressure is ejected. The work done on compression is represented by the area under the p-v curve 1-2-3-4. It is almost completely converted into heat. In the case of dry compressing vacuum pumps not much of this heat can be lost to the cooled casing due to the low density of the gas. This results in high gas temperatures within the pump. Experiences with claw pumps show that the highest temperatures occur at the rotors.

Shown in Fig. 2.27 is the principle of isochoric compression in a p-v diagram. Here the compression is not performed by reducing the volume of the pumping chamber, but by venting with cold gas which is applied from the outside after completion of the intake process. This is similar to the admission of a gas ballast when opening the gas ballast valve after completion of the intake phase. From the diagram it is apparent that in the case of isochoric compression the work done on compression must be increased, but cold gas instead of hot exhaust gas is used for venting. This method of direct gas cooling results in considerably reduced rotor temperatures. Pumps of this kind are discussed as "**ALL-ex**" in Section 2.1.3.2.2.

2.1.3.2.1 Claw pumps with internal compression for the semiconductor industry ("DRYVAC series")

Design of DRYVAC Pumps

Due to the work done on compression in the individual pumping stages, multi-stage claw pumps require water cooling for the four stages to remove the compression heat. Whereas the pumping chamber of the pump is free of sealants and lubricants, the gear and the lower pump shaft are lubricated

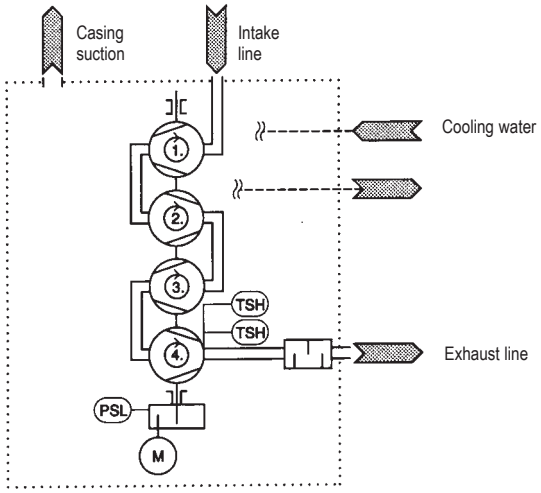


Fig. 2.29a Vacuum diagram for the DRYVAC B

with perfluoropolyether (PFPE). The gear box is virtually hermetically sealed from the pumping chamber by piston rings and a radial shaft seal. The bearings in the upper end disk are lubricated with PFPE grease. In order to protect the bearings and shaft seals against aggressive substances, a barrier gas facility is provided. A controlled water cooling system allows the control of the casing temperature over a wide range as the pump is subjected to differing gas loads coming from the process. The four stage design is available in several pumping speed and equipment grades of 25, 50 and 100 m^3/h DRYVAC pumps:

a) as the basic version for non-aggressive clean processes: DRYVAC 25 B, 50 B and 100 B (Fig. 2.29a)

b) as a version for semiconductor processes: DRYVAC 25 P, 50 P and 100 P (Fig. 2.29b)

c) as a system version with integrated self monitoring: DRYVAC 50 S and 100 S

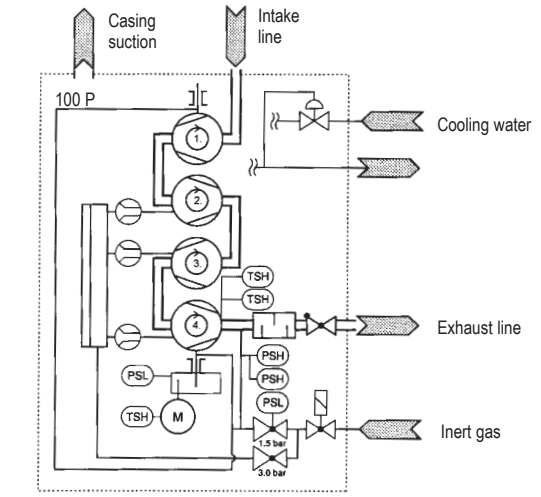


Fig. 2.29b Vacuum diagram for the DRYVAC P

Vacuum generation

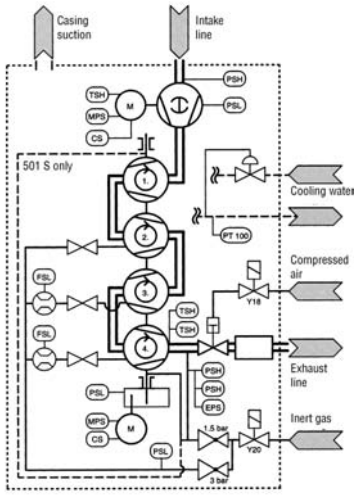


Fig. 2.29c Vacuum diagram for the DRYVAC S

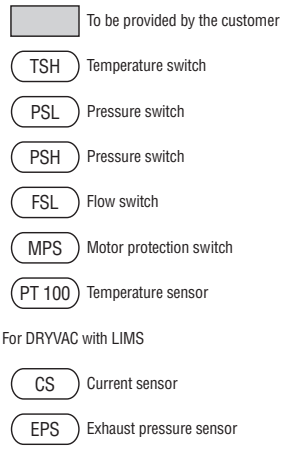


Fig. 2.30 Key to Figures 2.29a – 2.29c

d) as a system version with integrated self monitoring offering an increased pumping speed in the lower pressure range: DRYVAC 251 S and 501 S (Fig. 2.29c)

The ultimate pressure attainable with the DRYVAC 251 S or 501 S is – compared to the versions without integrated Roots pump – by approximately one order of magnitude lower (from $2 \cdot 10^{-2}$ mbar to $3 \cdot 10^{-3}$ mbar) and the attainable throughput is also considerably increased. It is of course possible to directly flange mount LEYBOLD RUVAC pumps on to the DRYVAC models (in the case of semiconductor processes also mostly with a PFPE oil filling for the bearing chambers).

The pumps of the DRYVAC family are the classic dry compressing claw vacuum pumps that are preferably used in the semiconductor industry, whereby the pumps need to meet a variety of special requirements. In semiconductor processes, as in many other vacuum applications, the formation of particles and dusts during the process and/or in the course of compressing the pumped substances to atmospheric pressure within the pump, is unavoidable. In the case of vacuum pumps operating on the claw principle it is possible to convey particles through the pump by means of so called “pneumatic conveying”. This prevents the deposition of particles and thus the formation of layers within the pump and reduces the risk that the claw rotor may seize. Care must be taken to ensure that the velocity of the gas flow within the individual pumping stages is at all times greater than the settling speed of the particles entrained in the gas flow. As can be seen in Fig. 2.31, the settling speed of the particles depends strongly on their size. The mean velocity (V_{Gas}) of the flowing gas during the compression phase is given by the following equation:

$$v_{Gas} = \frac{q_{pV}}{p \cdot A} \cdot \frac{mbar \cdot \ell \cdot s^{-1}}{mbar \cdot cm^2} = \frac{10 \cdot q_{pV}}{p \cdot A} \cdot \frac{m}{s} \qquad (2.22)$$

q_{pV} = gas throughput
p = pressure
A = surface area

One can see that with increasing pressure the velocity of the pumped gas flow slows down and attains the order of magnitude of the settling speed of the particles in the gas flow (Fig. 2.32). This means that the risk of

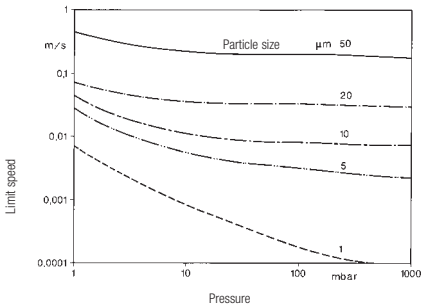


Fig. 2.31 Settling speed as a function of pressure p. Parameter: particle size

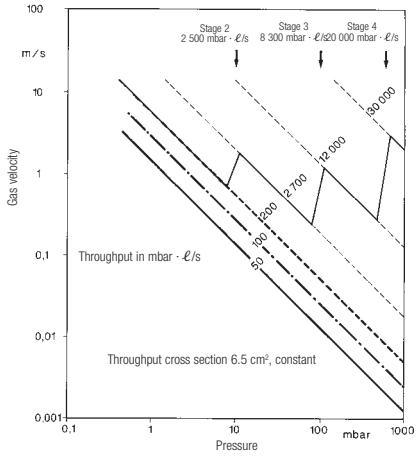


Fig. 2.32 Mean gas velocity v_g during compression without purge gas (left) and with purge gas (right) in stages 2, 3 and 4

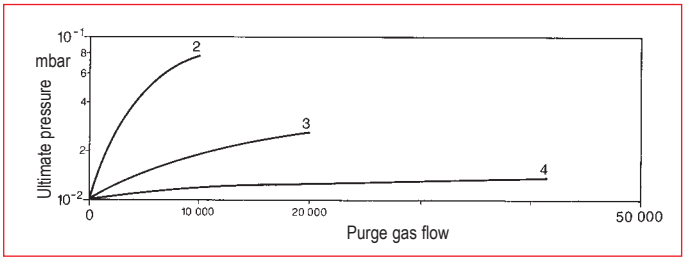


Fig. 2.33 Ultimate pressure of the DRYVAC 100S as a function of pure gas flow in stages 2 – 4

depositing particles in the operating chamber of the pump and the resulting impairment increases with increasing pressure. In parallel to this the potential for the formation of particles from the gaseous phase increases at increasing compression levels. In order to keep the size of the forming particles small and thus their settling speed low and to maintain a high velocity for the gas, an additional quantity of gas is supplied into the pump via the individual intermediate discs (purge gas). For this, the inflowing quantity of purge gas is matched to the pressure conditions prevailing in the individual pumping stages (see top right part of Fig. 2.32). This keeps the velocity of the gas flow high enough within the entire pump by so-called pneumatic pumping. Through the way in which the gas is lead within the pump, i.e. from the intake through the four pumping stages with the related intermediate discs to the exhaust, it is possible to reduce the influence of the purge gas on the ultimate pressure to a minimum. Test results (Fig. 2.33) indicate that the influence of purge gas in the fourth stage is – as to be expected – of the lowest level since there are located between this stage and the intake side the three other pumping stages. The admission of purge gas via the second and third stages (Fig. 2.33) has a comparatively small influence on the ultimate pressure as can be seen from the pumping speed curve in Fig. 2.34. Finally it can be said that the formation of particles is to be expected in most CVD processes. When using dry compressing claws vacuum pumps, the controlled admission of purge gas via the individual intermediate discs is the best approach to avoid the formation of layers. When applying this method several effects can be noted:

- The admitted purge gas dilutes the pumped mixture of substances, particle-forming reactions will not occur, or are at least delayed.
- The risk of an explosion through self-igniting substances is significantly reduced.
- Particles which have formed are conveyed pneumatically through the pump

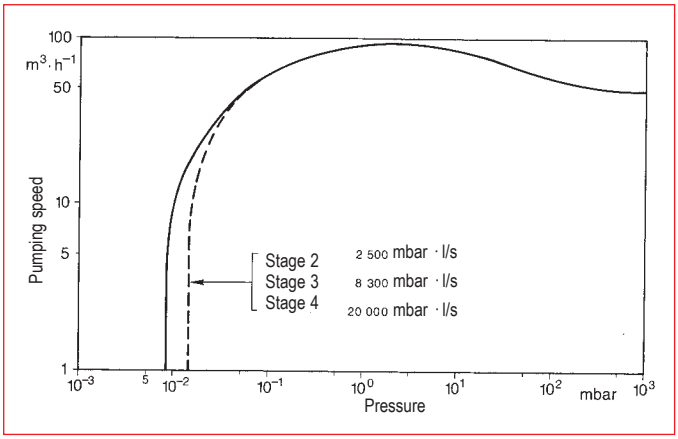


Fig. 2.34 Pumping speed with and without purge gas

- Losses in pumping speed and a reduction in ultimate pressure can be kept very small due to the special way in which the gas is made to pass through the pump.

2.1.3.2.2 Claw pumps without internal compression for chemistry applications ("ALL·ex")

The chemical industry requires vacuum pumps which are highly reliable and which do not produce waste materials such as contaminated waste oil or waste water. If this can be done, the operating costs of such a vacuum pump are low in view of the measures otherwise required for protecting the environment (disposal of waste oil and water, for example). For operation of the simple and rugged "ALL·ex" pump from LEYBOLD there are no restrictions as to the vapor flow or the pressure range during continuous operation. The "ALL·ex" may be operated within the entire pressure range from 5 to 1000 mbar without restrictions.

Design of the "ALL·ex" pump

The design of the two-stage ALL·ex is shown schematically in Fig. 2.35. The gas flows from top to bottom through the vertically arranged pumping stages in order to facilitate the ejection of condensates and rinsing liquids which may have formed. The casing of the pump is water cooled and permits cooling of the first stage. There is no sealed link between gas chamber and cooling channel so that the entry of cooling water into the pumping chamber can be excluded. The pressure-burst resistant design of the entire unit underlines the safety concept in view of protection against internal explosions, something which was also taken into account by direct cooling with cold gas (see operating principle). A special feature of the "ALL·ex" is that both shafts have their bearings exclusively in the gear. On the pumping side, the shafts are free (cantilevered). This simple design allows the user to quickly disassemble the pump for cleaning and servicing without the need for special tools.

In order to ensure a proper seal against the process medium in the pumping chamber the shaft seal is of the axial face seal type – a sealing

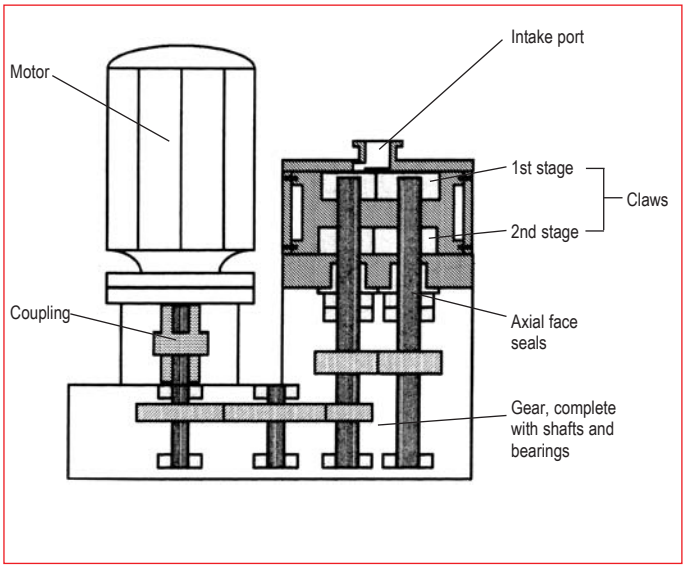
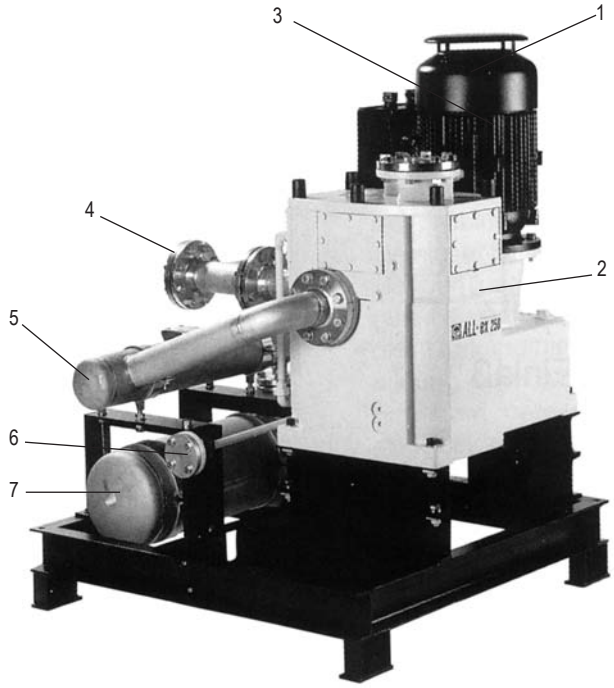


Fig. 2.35 Simple arrangement of the dry compression "ALL·ex" pump

Vacuum generation



1 Motor
2 Pump
3 Intake port
4 Exhaust port
5 Exhaust cooler
6 Cooling water connection
7 Cooling receiver

Fig. 2.36 "ALL·ex" pump

concept well proven in chemistry applications. This type of seal is capable of sealing liquids against liquids, so that the pump becomes rinseable and insensitive to forming condensate. Fig. 2.36 shows the components supplied with the ALL.ex, together with a gas cooler and a receiver.

Operating principle

Isochoric compression, which also serves the purpose of limiting the temperature ultimately attained during compression, especially in the stage on the side of the atmosphere, and which ensures protection against internal explosions, is performed by venting the pumping chamber with cold gas from a closed refrigerating gas cycle (Fig. 2.37). Fig. 2.38-1 indicates the start of the intake process by opening the intake slot through the control edge of the right rotor. The process gas then flows into the intake chamber which increases in size. The intake process is caused by the pressure gradient produced by increasing the volume of the pumping chamber. The maximum volume is attained after 3/4 of a revolution of the rotors (Fig. 2.38-2). After the end of the intake process, the control edge of the left rotor opens the cold gas inlet and at the same time the control edge of the right rotor opens the intake slot (Fig. 2.38-3) once more. In Fig. 2.38-4 the control edge of the left rotor terminates the discharge of the gas which has been compressed to 1000 mbar with the cold gas; at the same time the control edge of the right rotor completes an intake process again.

The total emissions from the system are not increased by the large quantities of cold gas, since a closed refrigerating cycle is maintained by way of an externally arranged gas cooler and condenser (Fig. 2.37). The hot exhaust gas is made to pass through the cooler and is partly returned in the form of cold gas for pre-admission cooling into the pump. The pump takes in the quantity of cold process gas needed for venting the pumping chamber back into the compression space on its own. This process, however, has no influence on the pumping speed of the "ALL·ex" because the intake process has already ended when the venting process starts. Designing the cooler as a condenser allows for simple solvent recovery. The method of direct gas cooling, i.e. venting of the pumping chamber with cold gas supplied from outside (instead of hot exhaust gas) results in the case of the "ALL·ex" in rotor temperatures which are so low that mixtures of substances rated as ExT3 can be pumped reliably under all operating conditions. The "ALL·ex" thus fully meets the requirements of the chemical industry concerning the protection against internal explosions. A certain degree of liquid compatibility makes the "ALL·ex" rinseable, thus avoiding the formation of layers in the pump, for example, or the capability of dissolving layers which may already have formed respectively. The rinsing liquids are usually applied to the pump after completion of the connected process (batch operation) or while the process is in progress during brief blocking phases. Even while the "ALL·ex" is at standstill and while the pumping chamber is completely filled with liquid it is possible to start this pump up. Shown in Fig. 2.39 is the pumping speed characteristic of an "ALL·ex" 250. This pump has a nominal pumping speed of 250 m^3/h and an ultimate pressure of < 10 mbar. At 10 mbar it still has a pumping speed of 100 m^3/h. The continuous operating pressure of the pump may be as high as 1000 mbar; it consumes 13.5 kW of electric power.

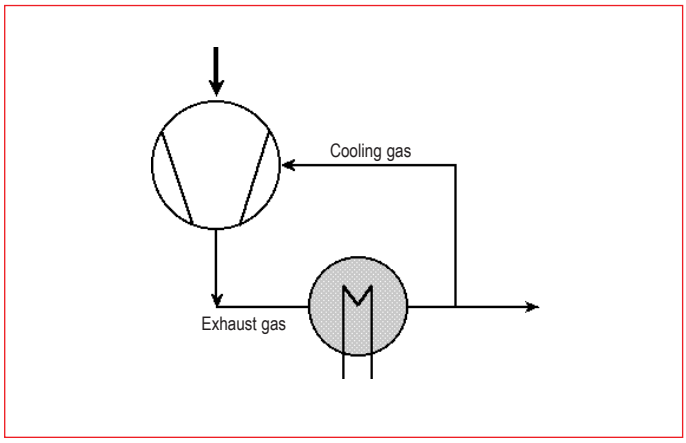


Fig. 2.37 Circulation of the cold gas in the “ALL·ex” with cooler / condenser

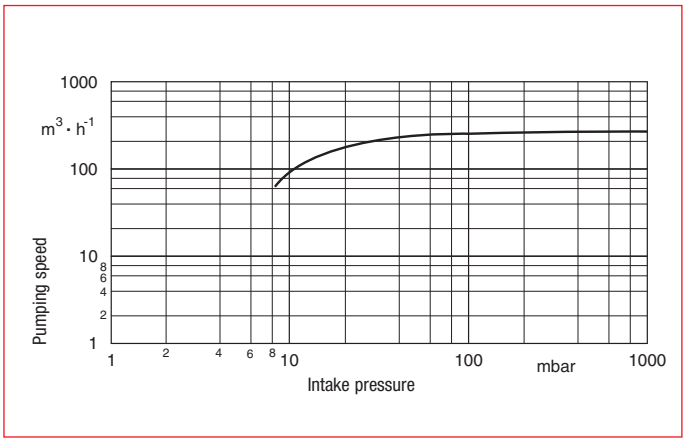


Fig. 2.39 Pumping speed characteristic of an ALL·ex 250

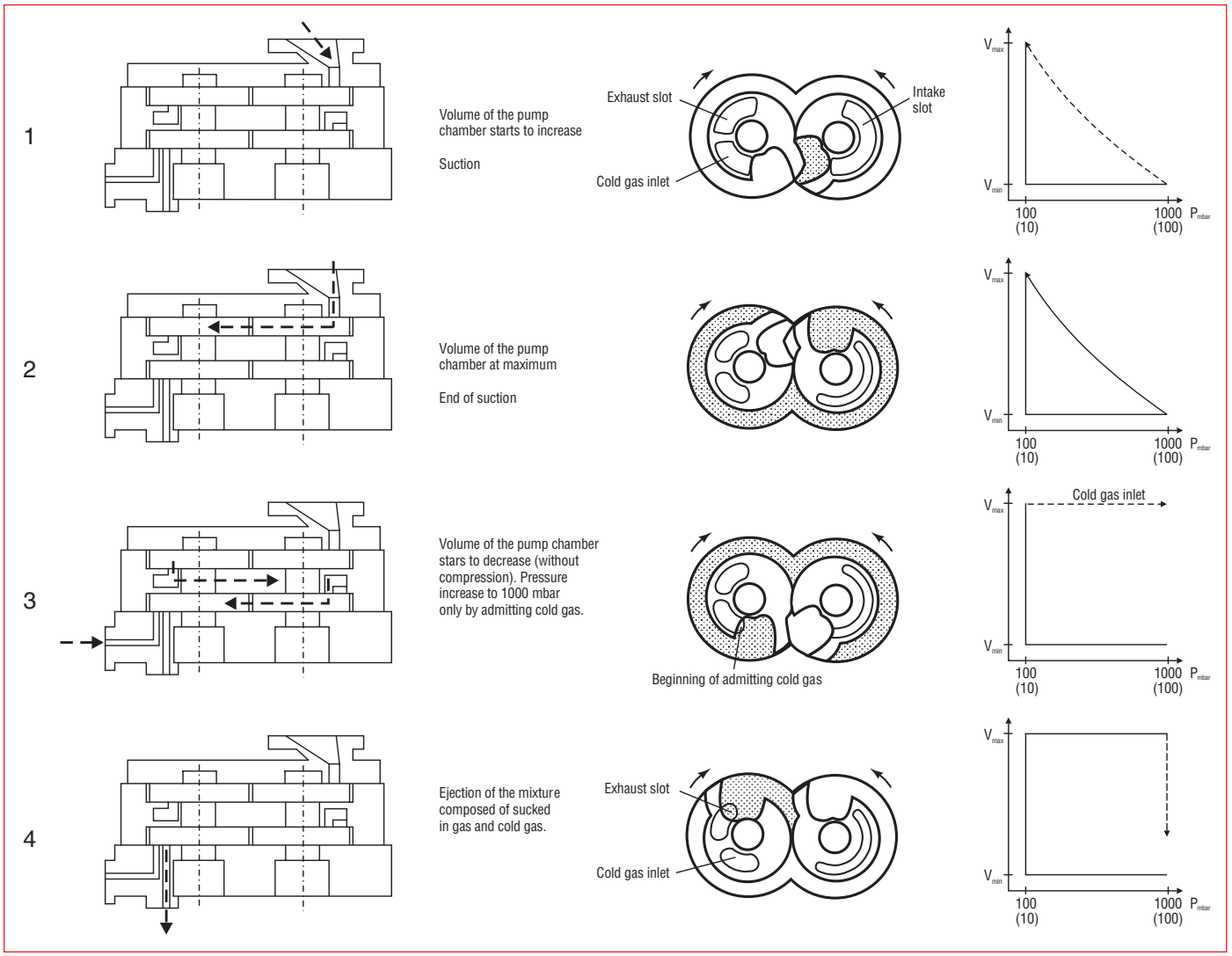


Fig. 2.38 Diagrams illustrating the pumping principle of the ALL·ex pump (claw pump without inner compression)

2.1.4 Accessories for oil-sealed rotary displacement pumps

During a vacuum process, substances harmful to rotary pumps can be present in a vacuum chamber.

Elimination of water vapor

Water vapor arises in wet vacuum processes. This can cause water to be deposited in the inlet line. If this condensate reaches the inlet port of the pump, contamination of the pump oil can result. The pumping performance of oil-sealed pumps can be significantly impaired in this way. Moreover, water vapor discharged through the outlet valve of the pump can condense in the discharge outlet line. The condensate can, if the outlet line is not correctly arranged, run down and reach the interior of the pump through the discharge outlet valve. Therefore, in the presence of water vapor and other vapors, the use of **condensate traps** is strongly recommended. If no discharge outlet line is connected to the gas ballast pump (e.g., with smaller rotary vane pumps), the use of **discharge filters** is recommended. These catch the oil mist discharged from the pump.

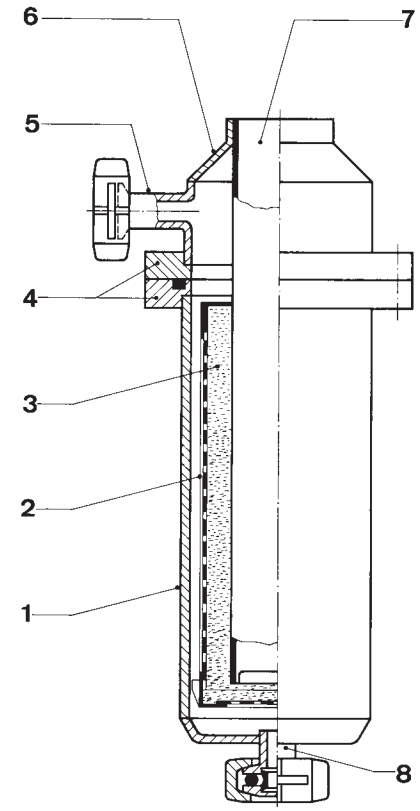
Some pumps have easily exchangeable filter cartridges that not only hold back oil mist, but clean the circulating pump oil. Whenever the amount of water vapor present is greater than the water vapor tolerance of the pump, a condenser should always be installed between the vessel and the pump. (For further details, see Section 2.1.5)

Elimination of dust

Solid impurities, such as dust and grit, significantly increase the wear on the pistons and the surfaces in the interior of the pump housing. If there is a danger that such impurities can enter the pump, a **dust separator** or a **dust filter** should be installed in the inlet line of the pump. Today not only conventional filters having fairly large casings and matching filter inserts are available, but also fine mesh filters which are mounted in the centering ring of the small flange. If required, it is recommended to widen the cross section with KF adaptors.

Elimination of oil vapor

The attainable ultimate pressure with oil-sealed rotary pumps is strongly influenced by water vapor and hydrocarbons from the pump oil. Even with two-stage rotary vane pumps, a small amount of back-streaming of these molecules from the pump interior into the vacuum chamber cannot be avoided. For the production of hydrocarbon-free high and ultrahigh vacuum, for example, with sputter-ion or turbomolecular pumps, a vacuum as free as possible of oil is also necessary on the forevacuum side of these pumps. To obtain this, **medium vacuum adsorption traps** (see Fig. 2.40) filled with a suitable adsorption material (e.g., LINDE molecular sieve 13X) are installed in the inlet line of such oil-sealed forepumps. The mode of action of a sorption trap is similar to that of an adsorption pump. For further details, see Section 2.1.8. If foreline adsorption traps are installed in the inlet line of oil-sealed rotary vane pumps in continuous operation, two adsorption traps in parallel are recommended, each separated by valves. Experience shows that the zeolite used as the adsorption material loses much of its adsorption capacity after about 10 – 14 days of running time, after which the other, now-regenerated, adsorption trap can be utilized; hence the process can continue uninterrupted. By heating the adsorption trap, which is now not connected in the pumping line, the vapors escaping



1 Housing
2 Basket holding the sieve
3 Molecular sieve (filling)
4 Sealing flanges
5 Intake port with small flange
6 Upper section
7 Vessel for the heater or refrigerant
8 Connection on the side of the pump with small flange

Fig. 2.40 Cross section of a medium vacuum adsorption trap

from the surface of the zeolite can be most conveniently pumped away with an auxiliary pump. In operation, pumping by the gas ballast pump generally leads to a covering of the zeolite in the other, unheated adsorption trap and thus to a premature reduction of the adsorption capacity of this trap.

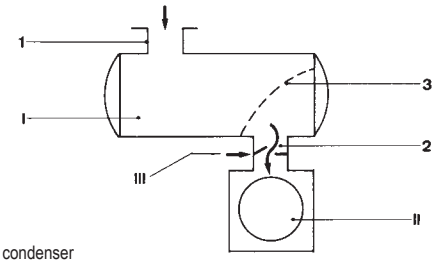
Reduction of the effective pumping speed

All filters, separators, condensers, and valves in the inlet line reduce the effective pumping speed of the pump. On the basis of the values of the conductances or resistances normally supplied by manufacturers, the actual pumping speed of the pump can be calculated. For further details, see Section 1.5.2.

2.1.5 Condensers

For pumping larger quantities of water vapor, the condenser is the most economical pump. As a rule, the condenser is cooled with water of such temperature that the condenser temperature lies sufficiently below the dew point of the water vapor and an economical condensation or pumping action is guaranteed. For cooling, however, media such as brine and refrigerants (NH_3, Freon) can also be used.

When pumping water vapor in a large industrial plant, a certain quantity of



1 Inlet of the condenser
2 Discharge of the condenser
3 See text

Fig. 2.41 Condenser (I) with downstream gas ballast pump (II) for pumping of large quantities of water vapor in the rough vacuum range (III) – adjustable throttle

air is always involved, which is either contained in the vapor or originates from leaks in the plant (the following considerations for air and water vapor obviously apply also in general for vapors other than water vapor). Therefore, the condenser must be backed by a gas ballast pump (see Fig. 2.41) and hence always works – like the Roots pump – in a combination. The gas ballast pump has the function of pumping the fraction of air, which is often only a small part of the water-vapor mixture concerned, without simultaneously pumping much water vapor. It is, therefore, understandable that, within the combination of condenser and gas ballast pump in the stationary condition, the ratios of flow, which occur in the region of rough vacuum, are not easily assessed without further consideration. The simple application of the continuity equation is not adequate because one is no longer concerned with a source or sink-free field of flow (the condenser is, on the basis of condensation processes, a sink). This is emphasized especially at this point. In a practical case of "non-functioning" of the condenser – gas ballast pump combination, it might be unjustifiable to blame the condenser for the failure.

In sizing the combination of condenser and gas ballast pump, the following points must be considered:

a) the fraction of permanent gases (air) pumped simultaneously with the water vapor should not be too great. At partial pressures of air that are more than about 5 % of the total pressure at the exit of the condenser, a marked accumulation of air is produced in front of the condenser surfaces. The condenser then cannot reach its full capacity (See also the account in Section 2.2.3 on the simultaneous pumping of gases and vapors).

b) the water vapor pressure at the condenser exit – that is, at the inlet side of the gas ballast pump – should not (when the quantity of permanent gas described in more detail in Section 2.2.3 is not pumped simultaneously) be greater than the water vapor tolerance for the gas ballast pump involved. If – as cannot always be avoided in practice – a higher water vapor partial pressure is to be expected at the condenser exit, it is convenient to insert a throttle between the condenser exit and the inlet port of the gas ballast pump. The conductance of this throttle should be variable and regulated (see Section 1.5.2) so that, with full throttling, the pressure at the inlet port of the gas ballast pump cannot become higher than the water vapor tolerance. Also, the use of other refrigerants or a decrease of the cooling water temperature may often permit the water vapor pressure to fall below the required value.

For a mathematical evaluation of the combination of condenser and gas ballast pump, it can be assumed that no loss of pressure occurs in the condenser, that the total pressure at the condenser entrance $p_{tot\,1}$, is equal to the total pressure at the condenser exit, $p_{tot\,2}$:

$$p_{tot\,1} = p_{tot\,2} \qquad (2.23)$$

The total pressure consists of the sum of the partial pressure portions of the air p_p and the water vapor p_v:

$$p_{p1} + p_{v1} = p_{p2} + p_{v2} \qquad (2.23a)$$

As a consequence of the action of the condenser, the water vapor pressure p_{D2} at the exit of the condenser is always lower than that at the entrance; for (2.23) to be fulfilled, the partial pressure of air p_{p2} at the exit must be higher than at the entrance p_{p1}, (see Fig. 2.43), even when no throttle is present.

The higher air partial pressure p_{p2} at the condenser exit is produced by an accumulation of air, which, as long as it is present at the exit, results in a stationary flow equilibrium. From this accumulation of air, the (eventually throttled) gas ballast pump in equilibrium removes just so much as streams from the entrance (1) through the condenser.

All calculations are based on (2.23a) for which, however, information on the quantity of pumped vapors and permanent gases, the composition, and the pressure should be available. The size of the condenser and gas ballast pump can be calculated, where these two quantities are, indeed, not mutually independent. Fig. 2.42 represents the result of such a calculation as an example of a condenser having a condensation surface of 1 m^2, and at an inlet pressure p_{v1}, of 40 mbar, a condensation capacity that amounts to 15 kg / h of pure water vapor if the fraction of the permanent gases is very small. 1 m^3 of cooling water is used per hour, at a line overpressure of 3 bar and a temperature of 12 °C. The necessary pumping speed of the gas ballast pump depends on the existing operating conditions, particularly the size of the condenser. Depending on the efficiency of the condenser, the water vapor partial pressure p_{v2} lies more or less above the saturation pressure p_S which corresponds to the temperature of the refrigerant. (By cooling with water at 12 °C, p_S, would be 15 mbar (see Table XIII in Section 9)). Correspondingly, the partial air pressure p_{p2} that prevails at the condenser exit also varies. With a large condenser, $p_{v2} \approx p_S$, the air partial pressure $p_{p,2}$ is thus large, and because $p_p \cdot V$ = const, the volume of air involved is small. Therefore, only a relatively small gas ballast pump is necessary. However, if the condenser is small, the opposite case arises:

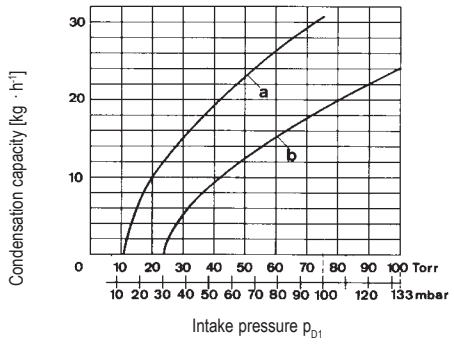


Fig. 2.42 Condensation capacity of the condenser (surface area available to condensation 1 m^2) as a function of intake pressure p_{D1} of the water vapor. Curve a: Cooling water temperature 12°C. Curve b: Temperature 25 °C. Consumption in both cases 1 m^3/h at 3 bar overpressure.

Vacuum generation

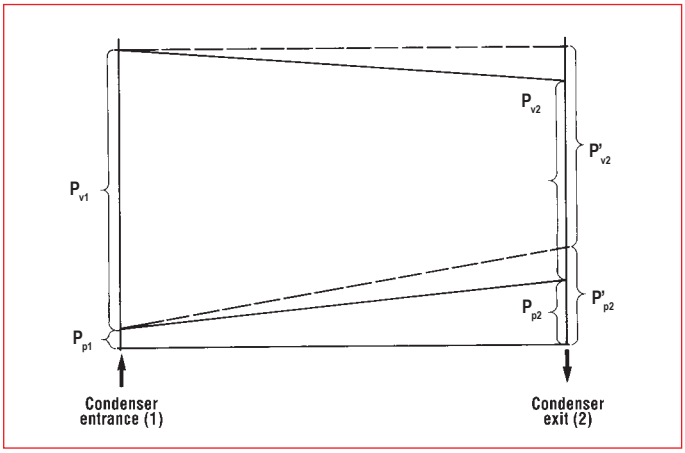


Fig. 2.43 Schematic representation of the pressure distribution in the condenser. The full lines correspond to the conditions in a condenser in which a small pressure drop takes place ($p_{tot\,2} < p_{tot\,1}$). The dashed lines are those for an ideal condenser ($p_{tot\,2} \approx p_{tot\,1}$). p_D: Partial pressure of the water vapor, p_L: Partial pressure of the air

$p_{v2} > p_S \cdot p_{p2}$, is small. Here a relatively large gas ballast pump is required. Since the quantity of air involved during a pumping process that uses condensers is not necessarily constant but alternates within more or less wide limits, the considerations to be made are more difficult. Therefore, it is necessary that the pumping speed of the gas ballast pump effective at the condenser can be regulated within certain limits.

In practice, the following measures are usual:

a) A **throttle section** is placed between the gas ballast pump and the condenser, which can be short-circuited during rough pumping. The flow resistance of the throttle section must be adjustable so that the effective speed of the pump can be reduced to the required value. This value can be calculated using the equations given in Section 2.2.3.

b) **Next to the large pump** for rough pumping a **holding pump with low speed** is installed, which is of a size corresponding to the minimum prevailing gas quantity. The objective of this holding pump is merely to maintain optimum operating pressure during the process.

c) The **necessary quantity of air** is admitted into the inlet line of the pump through a variable-leak valve. This additional quantity of air acts like an enlarged gas ballast, increasing the water vapor tolerance of the pump. However, this measure usually results in reduced condenser capacity. Moreover, the additional admitted quantity of air means additional power consumption and (see Section 8.3.1.1) increased oil consumption. As the efficiency of the condenser deteriorates with too great a partial pressure of air in the condenser, the admission of air should not be in front, but generally only behind the condenser.

If the starting time of a process is shorter than the total running time, technically the simplest method – the roughing and the holding pump – is used. Processes with strongly varying conditions require an adjustable throttle section and, if needed, an adjustable air admittance.

On the inlet side of the gas ballast pump a water vapor partial pressure p_{v2} is always present, which is at least as large as the saturation vapor pressure of water at the coolant temperature. This ideal case is realizable in practice only with a very large condenser (see above).

With a view to practice and from the stated fundamental rules, consider the two following cases:

1. **Pumping of permanent gases** with small amounts of water vapor. Here the size of the condenser – gas ballast pump combination is decided on the basis of the pumped-off permanent gas quantity. The condenser function is merely to reduce the water vapor pressure at the inlet port of the gas ballast pump to a value below the water vapor tolerance.

2. **Pumping of water vapor** with small amounts of permanent gases. Here, to make the condenser highly effective, as small as possible a partial pressure of the permanent gases in the condenser is sought. Even if the water vapor partial pressure in the condenser should be greater than the water vapor tolerance of the gas ballast pump, a relatively small gas ballast pump is, in general, sufficient with the then required throttling to pump away the prevailing permanent gases.

Important note: During the process, if the pressure in the condenser drops below the saturation vapor pressure of the condensate (dependent on the cooling water temperature), the condenser must be blocked out or at least the collected condensate isolated. If this is not done, the gas ballast pump again will pump out the vapor previously condensed in the condenser

2.1.6 Fluid-entrainment pumps

Basically, a distinction is made between ejector pumps such as water jet pumps (17 mbar < p < 1013 mbar), vapor ejector vacuum pumps (10^{-3} mbar < p < 10^{-1} mbar) and diffusion pumps (p < 10^{-3} mbar). Ejector vacuum pumps are used mainly for the production of medium vacuum. Diffusion pumps produce high and ultrahigh vacuum. Both types operate with a fast-moving stream of pump fluid in vapor or liquid form (water jet as well as water vapor, oil or mercury vapor). The pumping mechanism of all fluid-entrainment pumps is basically the same. The pumped gas molecules are removed from the vessel and enter into the pump fluid stream which expands after passing through a nozzle. The molecules of the pump fluid stream transfer by way of impact impulses to the gas molecules in the direction of the flow. Thus the gas which is to be pumped is moved to a space having a higher pressure.

In fluid-entrainment pumps corresponding vapor pressures arise during operation depending on the type of pump fluid and the temperature as well as the design of the nozzle. In the case of oil diffusion pumps this may amount to 1 mbar in the boiling chamber. The backing pressure in the pump must be low enough to allow the vapor to flow out. To ensure this, such pumps require corresponding backing pumps, mostly of the mechanical type. The vapor jet cannot enter the vessel since it condenses at the cooled outer walls of the pump after having been ejected through the nozzle.

Wolfgang Gaede was the first to realize that gases at comparatively low pressure can be pumped with the aid of a pump fluid stream of essentially higher pressure and that, therefore, the gas molecules from a region of low total pressure move into a region of high total pressure. This apparently paradoxical state of affairs develops as the vapor stream is initially entirely free of gas, so that the gases from a region of higher partial gas pressure (the vessel) can diffuse into a region of lower partial gas pressure (the vapor stream). This basic Gaede concept was used by Langmuir (1915) in the construction of the first modern diffusion pump. The first diffusion pumps were mercury diffusion pumps made of glass, later of metal. In the Sixties, mercury as the medium was almost completely replaced by oil. To

obtain as high a vapor stream velocity as possible, he allowed the vapor stream to emanate from a nozzle with supersonic speed. The pump fluid vapor, which constitutes the vapor jet, is condensed at the cooled wall of the pump housing, whereas the transported gas is further compressed, usually in one or more succeeding stages, before it is removed by the backing pump. The compression ratios, which can be obtained with fluid entrainment pumps, are very high: if there is a pressure of 10^{-9} mbar at the inlet port of the fluid-entrainment pump and a backing pressure of 10^{-2} mbar, the pumped gas is compressed by a factor of 10^{7}!

Basically the ultimate pressure of fluid entrainment pumps is restricted by the value for the partial pressure of the fluid used at the operating temperature of the pump. In practice one tries to improve this by introducing baffles or cold traps. These are “condensers” between fluid entrainment pump and vacuum chamber, so that the ultimate pressure which can be attained in the vacuum chamber is now only limited by the partial pressure of the fluid at the temperature of the baffle.

The various types of fluid entrainment pumps are essentially distinguished by the density of the pump fluid at the exit of the top nozzle facing the high vacuum side of the pump:

1. Low vapor density:
   Diffusion pumps

   Oil diffusion pumps
   (Series: LEYBODIFF, DI and DIP)
   Mercury diffusion pumps

2. High vapor density:
   Vapor jet pumps

   Water vapor pumps
   Oil vapor jet pumps
   Mercury vapor jet pumps

3. **Combined**
   oil diffusion/ vapor jet pumps

4. **Water jet pumps**

Cooling of fluid entrainment pumps

The heater power that is continuously supplied for vaporizing the pump fluid in fluid-entrainment pumps must be dissipated by efficient cooling. The energy required for pumping the gases and vapors is minimal. The outside walls of the casing of diffusion pumps are cooled, generally with water. Smaller oil diffusion pumps can, however, also be cooled with an air stream because a low wall temperature is not so decisive to the efficiency as for mercury diffusion pumps. Oil diffusion pumps can operate well with wall temperatures of 30 °C, whereas the walls of mercury diffusion pumps must be cooled to 15 °C. To protect the pumps from the danger of failure of the cooling water – insofar as the cooling-water coil is not controlled by thermally operated protective switching – a water circulation monitor should be installed in the cooling water circuit; hence, evaporation of the pump fluid from the pump walls is avoided.

2.1.6.1 (Oil) Diffusion pumps

These pumps consist basically (see Fig. 2.44) of a pump body (3) with a cooled wall (4) and a three- or four-stage nozzle system (A – D). The oil serving as pump fluid is in the boiler (2) and is vaporized from here by electrical heating (1). The pump fluid vapor streams through the riser tubes and emerges with supersonic speed from the ring-shaped nozzles (A – D). Thereafter the jet so-formed widens like an umbrella and reaches the wall where condensation of the pump fluid occurs. The liquid condensate flows downward as a thin film along the wall and finally returns into the boiler. Because of this spreading of the jet, the vapor density is relatively low. The diffusion of air or any pumped gases (or vapors) into the jet is so rapid that despite its high velocity the jet becomes virtually completely saturated with the pumped medium. Therefore, over a wide pressure range diffusion pumps have a high pumping speed. This is practically constant over the entire working region of the diffusion pump (≤ 10^{-3} mbar) because the air at these low pressures cannot influence the jet, so its course remains undisturbed. At higher inlet pressures, the course of the jet is altered. As a result, the pumping speed decreases until, at about 10^{-1} mbar, it becomes immeasurably small.

The forevacuum pressure also influences the vapor jet and becomes detrimental if its value exceeds a certain critical limit. This limit is called **maximum backing pressure or critical forepressure**. The capacity of

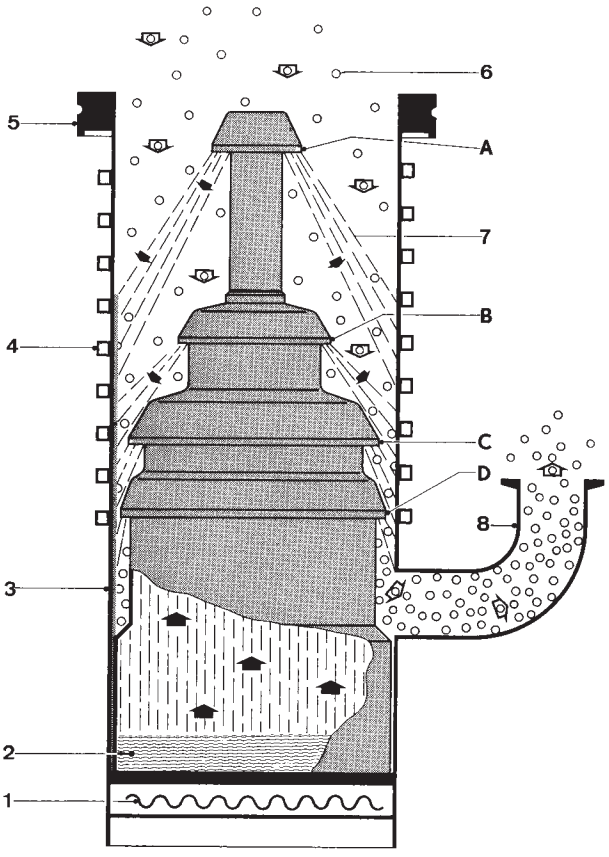


Fig. 2.44 Mode of operation of a diffusion pump

Vacuum generation

the chosen backing pump must be such (see 2.3.2) that the amount of gas discharged from the diffusion pump is pumped off without building up a backing pressure that is near the maximum backing pressure or even exceeding it.

The attainable ultimate pressure depends on the construction of the pump, the vapor pressure of the pump fluid used, the maximum possible condensation of the pump fluid, and the cleanliness of the vessel. Moreover, backstreaming of the pump fluid into the vessel should be reduced as far as possible by suitable baffles or cold traps (see Section 2.1.6.4).

Degassing of the pump oil

In oil diffusion pumps it is necessary for the pump fluid to be degassed before it is returned to the boiler. On heating of the pump oil, decomposition products can arise in the pump. Contamination from the vessel can get into the pump or be contained in the pump in the first place. These constituents of the pump fluid can significantly worsen the ultimate pressure attainable by a diffusion pump, if they are not kept away from the vessel. Therefore, the pump fluid must be freed of these impurities and from absorbed gases.

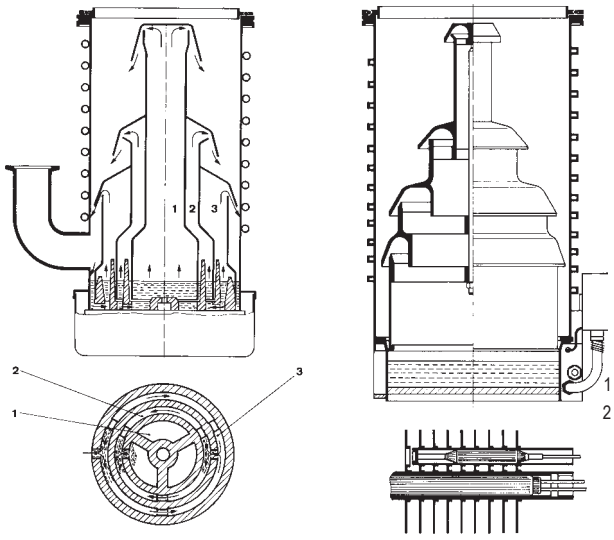
This is the function of the **degassing section**, through which the circulating oil passes shortly before re-entry into the boiler. In the degassing section, the most volatile impurities escape. Degassing is obtained by the carefully controlled temperature distribution in the pump. The condensed pump fluid, which runs down the cooled walls as a thin film, is raised to a temperature of about 130 °C below the lowest diffusion stage, to allow the volatile components to evaporate and be removed by the backing pump. Therefore, the re-evaporating pump fluid consists of only the less volatile components of the pump oil.

Pumping speed

The magnitude of the specific pumping speed S of a diffusion pump – that is, the pumping speed per unit of area of the actual inlet surface – depends on several parameters, including the position and dimensions of the high vacuum stage, the velocity of the pump fluid vapor, and the mean molecular velocity $\bar{c}$ of the gas being pumped (see equation 1.17 in Section 1.1). With the aid of the kinetic theory of gases, the maximum attainable specific pumping speed at room temperature on pumping air is calculated to $S_{max} = 11.6\ l \cdot s^{-1} \cdot cm^{-2}$. This is the specific (molecular) flow conductance of the intake area of the pump, resembling an aperture of the same surface area (see equation 1.30 in Section 1.5.3). Quite generally, diffusion pumps have a higher pumping speed for lighter gases compared to heavier gases.

To characterize the effectiveness of a diffusion pump, the so called HO factor is defined. This is the ratio of the actually obtained specific pumping speed to the theoretical maximum possible specific pumping speed. In the case of diffusion pumps from LEYBOLD optimum values are attained (of 0.3 for the smallest and up to 0.55 for the larger pumps).

The various oil diffusion pumps manufactured by LEYBOLD differ in the following design features (see Fig. 2.45 a and b).



a) LEYBODIFF-Pump with fractionating facility
1 Center
2 Middle section
3 Outer part of the boiler (fractionation)

b) DI pump; side view on to the internal heater
1 Thermostat sensor
2 Heating cartridge

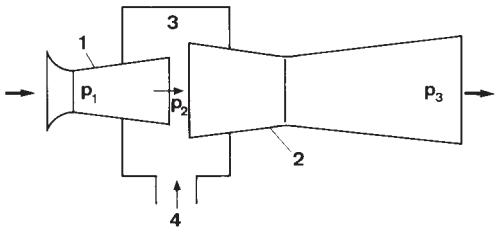
Fig. 2.45 Diagram showing the basic differences in LEYBOLD oil diffusion pumps

a) **LEYBODIFF series**

This series of pumps is equipped with a **fractionating device**. The various constituents of the pump fluid are selected so that the high vacuum nozzle is supplied only by the fraction of the pump fluid that has the lowest vapor pressure. This assures a particularly low ultimate pressure. Fractionating occurs because the degassed oil first enters the outer part of the boiler, which serves the nozzle on the backing vacuum side. Here a part of the more volatile constituents evaporates. Hence the already purified pump fluid reaches the intermediate part of the boiler, which serves the intermediate nozzle. Here the lighter constituents are evaporated in greater quantities than the heavier constituents. When the oil enters the central region of the boiler, which serves the high vacuum nozzle, it has already been freed of the light volatile constituents.

b) **DI series**

In these pumps an evaporation process for the pump fluid which is essentially free of bursts is attained by the exceptional heater design resulting in a highly constant pumping speed over time. The heater is of the internal type and consists of heating cartridges into which tubes with soldered on thermal conductivity panels are introduced. The tubes made of stainless steel are welded horizontally into the pump's body and are located above the oil level. The thermal conductivity panels made of copper are only in part immersed in the pump fluid. Those parts of the thermal conductivity panels are so rated that the pump fluid can evaporate intensively but without any retardation of boiling. Those parts of the thermal conductivity panels above the oil level supply additional energy to the vapor. Owing to the special design of the heating system, the heater cartridges may be exchanged also while the pump is still hot.

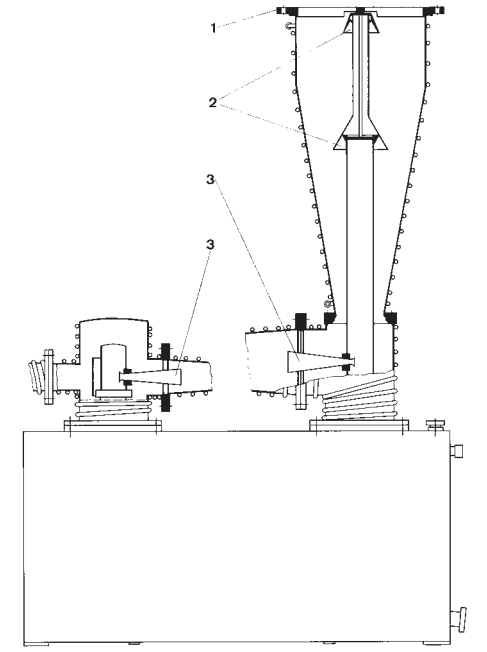


1 Nozzle (Laval)
2 Diffuser nozzle (Venturi)
3 Mixing chamber
4 Connection to the vacuum chamber

Fig. 2.46 Operation of a vapor jet pump

2.1.6.2 Oil vapor ejector pumps

The pumping action of a vapor ejector stage is explained with the aid of Fig. 2.46. The pump fluid enters under high pressure p_1 the nozzle (1), constructed as a Laval nozzle. There it is **expanded** to the inlet pressure p_2. On this expansion, the sudden change of energy is accompanied by an increase of the velocity. The consequently accelerated pump fluid vapor jet streams through the mixer region (3), which is connected to the vessel (4) being evacuated. Here the gas molecules emerging from the vessel are dragged along with the vapor jet. The mixture, pump fluid vapor – gas, now enters the diffuser nozzle constructed as a Venturi nozzle (2). Here the vapor – gas mixture is **compressed** to the backing pressure p_3 with simultaneous diminution of the velocity. The pump fluid vapor is then condensed at the pump walls, whereas the entrained gas is removed by the backing pump. Oil vapor ejector pumps are ideally suited for the pumping of larger quantities of gas or vapor in the pressure region between 1 and 10^{-3} mbar. The higher density of the vapor stream in the nozzles ensures that the diffusion of the pumped gas in the vapor stream takes place much more slowly than in diffusion pumps, so that only the outer layers of the vapor stream are permeated by gas. Moreover, the surface through which the diffusion occurs is much smaller because of the special construction of the nozzles. The specific pumping speed of the vapor ejector pumps is, therefore, smaller than that of the diffusion pumps. As the pumped gas in the neighborhood of the jet under the essentially higher inlet pressure decisively influences the course of the flow lines, optimum conditions are obtained only at certain inlet pressures. Therefore, the pumping speed



1 High vacuum port 2 Diffusion stages 3 Ejector stages

Fig. 2.47 Diagram of an oil jet (booster) pump

does not remain constant toward low inlet pressures. As a consequence of the high vapor stream velocity and density, oil vapor ejector pumps can transport gases against a relatively high backing pressure. Their critical backing pressure lies at a few millibars. The oil vapor ejector pumps used in present-day vacuum technology have, in general, one or more diffusion stages and several subsequent ejector stages. The nozzle system of the booster is constructed from two diffusion stages and two ejector stages in cascade (see Fig. 2.47). The diffusion stages provide the high pumping speed between 10^{-4} and 10^{-3} mbar (see Fig. 2.48), the ejector stages, the high gas throughput at high pressures (see Fig. 2.49) and the high critical backing pressure. Insensitivity to dust and vapors dissolved in the pump fluid is obtained by a spacious boiler and a large pump fluid reservoir. Large quantities of impurities can be contained in the boiler without deterioration of the pumping characteristics.

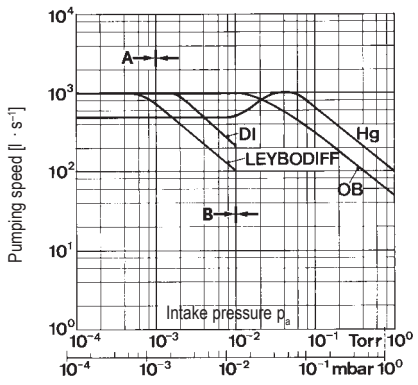


Fig. 2.48 Pumping speed of various vapor pumps as a function of intake pressure related to a nominal pumping speed of 1000 l/s.
End of the working range of oil vapor ejector pumps (A) and diffusion pumps (B)

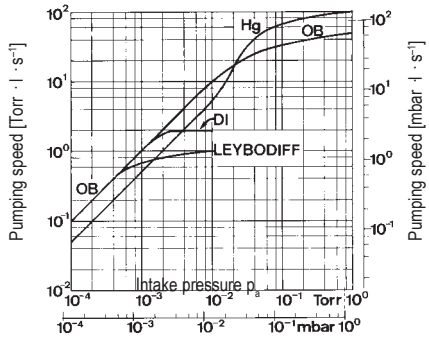


Fig. 2.49 Pumping speed of various vapor pumps (derived from Fig. 2.48)

2.1.6.3 Pump fluids

a) Oils

The suitable pump fluids for oil diffusion pumps are mineral oils, silicone oils, and oils based on the polyphenyl ethers. Severe demands are placed on such oils which are met only by special fluids. The properties of these, such as vapor pressure, thermal and chemical resistance, particularly against air, determine the choice of oil to be used in a given type of pump or to attain a given ultimate vacuum. The vapor pressure of the oils used in vapor pumps is lower than that of mercury. Organic pump fluids are more sensitive in operation than mercury, because the oils can be decomposed by long-term admission of air. Silicone oils, however, withstand longer lasting frequent admissions of air into the operational pump.

Typical mineral oils are **DIFFELEN light, normal** and **ultra**. The different types of DIFFELEN are close tolerance fractions of a high quality base product (see our catalog).

Silicone oils (**DC 704, DC 705**, for example) are uniform chemical compounds (organic polymers). They are highly resistant to oxidation in the case of air inrushes and offer special thermal stability characteristics.

DC 705 has an extremely low vapor pressure and is thus suited for use in diffusion pumps which are used to attain extremely low ultimate pressures of $< 10^{-10}$ mbar.

ULTRALEN is a polyphenylether. This fluid is recommended in all those cases where a particularly oxidation-resistant pump fluid must be used and where silicone oils would interfere with the process.

APIEZON AP 201 is an oil of exceptional thermal and chemical resistance capable of delivering the required high pumping speed in connection with oil vapor ejector pumps operating in the medium vacuum range. The attainable ultimate total pressure amounts to about 10^{-4} mbar.

b) Mercury

Mercury is a very suitable pump fluid. It is a chemical element that during vaporization neither decomposes nor becomes strongly oxidized when air is admitted. However, at room temperature it has a comparatively high vapor pressure of 10^{-3} mbar. If lower ultimate total pressures are to be reached, cold traps with liquid nitrogen are needed. With their aid, ultimate total pressures of 10^{-10} mbar can be obtained with mercury diffusion pumps. Because mercury is toxic, as already mentioned, and because it presents a hazard to the environment, it is nowadays hardly ever used as a pump fluid. LEYBOLD supplies pumps with mercury as the pump fluid only on request. The vapor pressure curves of pump fluids are given in Fig. 9.12, Section 9.

2.1.6.4 Pump fluid backstreaming and its suppression (vapor barriers, baffles)

In the vapor stream from the topmost nozzle of a diffusion pump, pump fluid molecules not only travel in the direction of streaming to the cooled pump wall, but also have backward components of velocity because of intermolecular collisions. They can thus stream in the direction of the vessel. In the case of LEYBODIFF and DI pumps, the oil-backstreaming amounts to a few micrograms per minute for each square centimeter of inlet cross-sectional area. To reduce this backstreaming as much as possible, various measures must be undertaken simultaneously:

a) the high vacuum-side nozzle and the shape of the part of the pump body surrounding this nozzle must be constructed so that as few as possible vapor molecules emerge sideways in the path of the vapor stream from the nozzle exit to the cooled pump wall.

b) the method for cooling the pump wall must allow as complete as possible condensation of the pump fluid vapor and, after condensation, the fluid must be able to drain away readily.

c) one or more pump-fluid traps, baffles, or cold traps must be inserted between the pump and the vessel, depending on the ultimate pressure that is required.

Two chief requirements must be met in the construction of baffles or cold traps for oil diffusion pumps. First, as far as possible, all backstreaming pump-fluid vapor molecules should remain attached to (condensed at) the inner cooled surfaces of these devices. Second, the condensation surfaces must be so constructed and geometrically arranged that the flow conductance of the baffles or cold traps is as large as possible for the pumped gas. These two requirements are summarized by the term “optically opaque”. This means that the particles cannot enter the baffle without hitting the wall, although the baffle has a high conductance. The implementation of this idea has resulted in a variety of designs that take into account one or the other requirement.

A **cold cap baffle** is constructed so that it can be mounted immediately above the high vacuum nozzle. The cold cap baffle is made of metal of high thermal conductivity in good thermal contact with the cooled pump wall, so that in practice it is maintained at the cooling-water temperature or, with air-cooled diffusion pumps, at ambient temperature. In larger types of pumps, the cold cap baffle is water cooled and permanently attached to the pump body. The effective pumping speed of a diffusion pump is reduced by about 10 % on installation of the cold cap baffle, but the oil backstreaming is reduced by about 90 to 95 %.

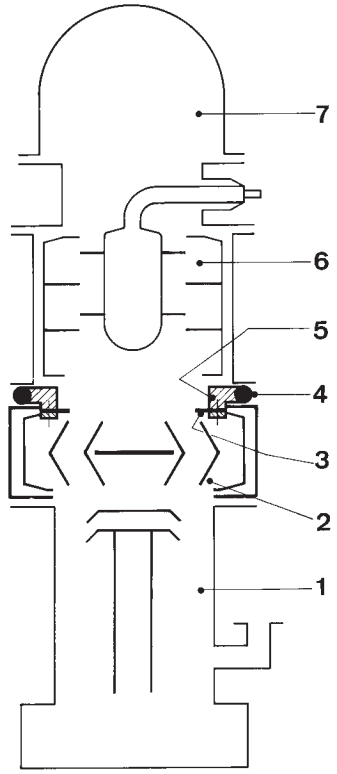
Shell baffles consist of concentrically arranged shells and a central baffle plate. With appropriate cooling by water or refrigeration, almost entirely oil vapor-free vacua can be produced by this means. The effective pumping speed of the diffusion pump remains at least at 50 %, although shell baffles are optically opaque. This type of baffle has been developed by LEYBOLD in two different forms: with a stainless-steel cooling coil or – in the so-called Astrotorus baffles – with cooling inserts of copper. The casing of the former type is made entirely of stainless steel.

For the smaller air-cooled, oil diffusion pumps, **plate baffles** are used. The air-cooled arrangement consists of a copper plate with copper webs to the housing wall. The temperature of the plate baffle remains nearly ambient during the operation of the diffusion pump.

Hydrocarbon-free vacuum

If extreme demands are made on freedom from oil vapor with vacuum produced by oil diffusion pumps, **cold traps** should be used that are cooled with liquid nitrogen so that they are maintained at a temperature of - 196 °C.

Low-temperature baffles or cold traps should always be used with a cold cap in place. On this the greatest part of the backstreaming oil is condensed, so that the inevitable loss of pump fluid from the condensation of the pump fluid on the low-temperature surface is kept at a minimum.



1 Diffusion pump with cold cap baffle (cooled by contact),
2 Shell or chevron baffle
3 Anticreep barrier
4 Sealing gasket
5 Bearing ring
6 LN2 cold trap,
7 Vacuum chamber

Fig. 2.50 Schematic arrangement of baffle, anticreep barrier and cold trap above a diffusion pump

With longer-term operation it is always advisable to install, in place of the cold cap, a water-cooled shell or chevron baffle between the diffusion pump and the low-temperature baffle or cold trap (see Fig. 2.50).

LEYBOLD manufactures cold traps made of metal so called **LN_2 cold traps**. These cold traps are to be used in those cases where a cold trap is to be operated for prolonged periods of time without requiring a filling facility for liquid nitrogen. The temperature increase at the vessel containing the refrigerant is so slight over the operating time that – as the liquid level drops – no significant desorption of the condensate takes place. Located on the pumping side is an impact panel made of copper. The low temperature of this panel ensures that the greater part of the condensed pump fluid remains in the liquid state and may drip back into the pump. Today the oils used to operate diffusion pumps have a very low vapor pressure at room temperature (for example DIFFELEN light, $2 \cdot 10^{-8}$ mbar; DC 705, $4 \cdot 10^{-10}$ mbar). The specified provisions with a liquid-nitrogen-cooled baffle or cold trap would enable an absolutely oil-free vacuum to be produced.

In practice, however, complete suppression of oil-backstreaming is never attained. There are always a few pump-fluid molecules that, as a result of collisions with one another, reach the vessel without having hit one of the cooled surfaces of the baffle or the cold trap. Moreover, there are always a few highly volatile components of the pump fluid that do not remain attached to the very low temperature surfaces. The temperature and the vapor molecules adsorbed at the surface of the vessel determine exactly the pressure in the vessel. If, the surfaces are not fully covered with adsorbed pump-fluid molecules after a bake-out process, their vapor pressure contributes only insignificantly to the pressure in the vessel.

After a certain time, the “stay-down time”, a continuous layer of oil molecules builds up, and the ultimate pressure is practically determined by the vapor pressure of the pump fluid at the temperature of the vessel walls. This “stay-down” time can even amount to several hours, indeed even to days, with the use of low-temperature baffles.

Oil can reach the vessel not only as vapor, but also as a liquid film, because oil wets readily and thus creeps up the wall.

By installation of an **anticreep barrier** (see Fig. 2.50) made of Teflon polymer, a material that is not wetted by oil and can stand a bake-out temperature up to 200 °C, further creeping of the oil can be effectively prevented. It is most appropriate to arrange the anticreep barrier above the upper baffle (see Fig. 2.50).

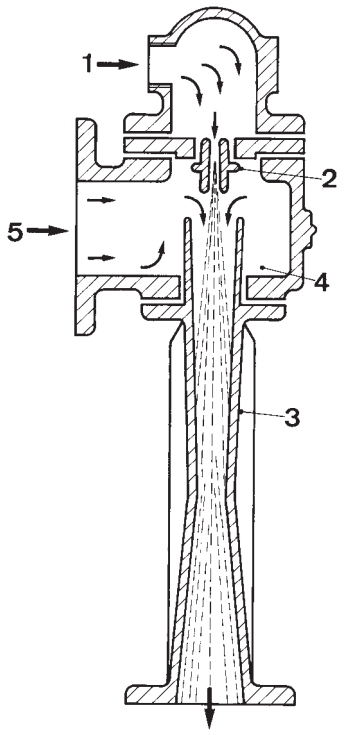
Note:
It must be noted that data on backstreaming as specified in catalogs apply only to continuously-operated oil diffusion pumps. Shortly after starting a pump the uppermost nozzle will not eject a well directed vapor jet. Instead oil vapor spreads in all directions for several seconds and the backstreaming effect is strong. When switching a diffusion pump on and off frequently the degree of oil brackstreaming will be greater.

2.1.6.5 Water jet pumps and steam ejectors

Included in the class of fluid-entrainment pumps are not only pumps that use a fast-streaming vapor as the pump fluid, but also liquid jet pumps. The simplest and cheapest vacuum pumps are **water jet pumps**. As in a vapor pump (see Fig. 2.46 or 2.51), the liquid stream is first released from a nozzle and then, because of turbulence, mixes with the pumped gas in the mixing chamber. Finally, the movement of the water – gas mixture is slowed down in a Venturi tube. The ultimate total pressure in a container that is pumped by a water jet pump is determined by the vapor pressure of the water and, for example, at a water temperature of 15 °C amounts to about 17 mbar.

Essentially higher pumping speeds and lower ultimate pressures are produced by **steam ejector pumps**. The section through one stage is shown in Fig. 2.51. The markings correspond with those shown in Fig. 2.46. In practice, several pumping stages are usually mounted in cascade. For laboratory work, two-stage pump combinations are suitable and consist of a steam ejector stage and a water jet (backing) stage, both made of glass. The water jet backing stage enables operation without other backing pumps. With the help of a vapor stream at overpressure, the vacuum chamber can be evacuated to an ultimate pressure of about 3 mbar. The condensate from the steam is led off through the drain attachment. The water jet stage of this pump is cooled with water to increase its efficiency. Steam ejector pumps are especially suitable for work in laboratories, particularly if very aggressive vapors are to be pumped. Steam ejector pumps, which will operate at a pressure of a few millibars, are especially recommended for pumping laboratory distillation apparatus and similar plants when the pressure from a simple water jet pump is insufficient. In this instance, the use of rotary pumps would not be economical.

Vacuum generation



1 Steam inlet
2 Jet nozzle
3 Diffuser
4 Mixing region
5 Connection to the vacuum chamber

Fig. 2.51 Schematic representation of the operation of a steam ejector pump

Even in spite of their low investment costs water jet pumps and steam ejectors are being replaced in the laboratories more and more by diaphragm pumps because of the environmental problems of using water as the pump fluid. Solvent entering the water can only be removed again through complex cleaning methods (distillation).

2.1.7 Turbomolecular pumps

The principle of the molecular pump – well known since 1913 – is that the gas particles to be pumped receive, through impact with the rapidly moving surfaces of a rotor, an impulse in a required flow direction. The surfaces of the rotor – usually disk-shaped – form, with the stationary surfaces of a stator, intervening spaces in which the gas is transported to the backing port. In the original **Gaede molecular pump** and its modifications, the intervening spaces (transport channels) were very narrow, which led to constructional difficulties and a high degree of susceptibility to mechanical contamination.

At the end of the Fifties, it became possible – through a turbine-like design and by modification of the ideas of Gaede – to produce a technically viable pump the socalled "**turbomolecular pump**". The spaces between the stator and the rotor disks were made in the order of millimeters, so that essentially larger tolerances could be obtained. Thereby, greater security in operation was achieved. However, a pumping effect of any significance is only attained when the circumferential velocity (at the outside rim) of the rotor blades reaches the order of magnitude of the average thermal velocity of the molecules which are to be pumped. Kinetic gas theory supplies for $\bar{c}$ othe equation 1.17:

$$\bar{c} = \sqrt{\frac{8 \cdot R \cdot T}{\pi \cdot M}}$$

in which the dependency on the type of gas as a function of molar mass M is contained. The calculation involving cgs-units (where R = 83.14 · 106 mbar · cm³ / mol · K) results in the following Table:

Gas	Molar Mass M	Mean thermal velocity (m/s)
H_2	2	1761
He	4	1245
H_2O	18	587
Ne	20	557
CO	28	471
N_2	28	471
Air	28.96	463
O_2	32	440
Ar	40	394
CO_2	44	375
$CC_{13}F$ (F11)	134.78	68

Table 2.4 cas a function of molar mass M

Whereas the dependence of the pumping speed on the type of gas is fairly low ($S \sim \bar{c} \sim 1 / \sqrt{M}$), the dependence of the compression k_0 at zero throughput and thus also the compression k, because of $k_0 \sim e^{\sqrt{M}}$ $\log k_0 \sim \sqrt{M}$, is greater as shown by the experimentally-determined relationship in Fig. 2.55.

Example:

from theory it follows that

$$\frac{\log k_0(He)}{\log k_0(N_2)} = \sqrt{\frac{4}{28}} = \sqrt{\frac{1}{7}} = \frac{1}{2.65}$$

$$\Rightarrow \log k_0(N_2) = 2.65 \cdot \log k_0(He)$$

this with k_0 (He) = 3 · 10³ from Fig. 2.55 results in:

$\log k_0 (N_2) = 2.65 \cdot \log (3 \cdot 10^3) = 9.21$

or $k_0 (N_2) = 1.6 \cdot 10^9$.

This agrees – as expected – well (order of magnitude) with the experimentally determined value for $k_0 (N_2) = 2.0 \cdot 10^8$ from Fig. 2.55. In view of the optimizations for the individual rotor stages common today, this consideration is no longer correct for the entire pump. Shown in Fig. 2.56 are the values as measured for a modern TURBOVAC 340 M.

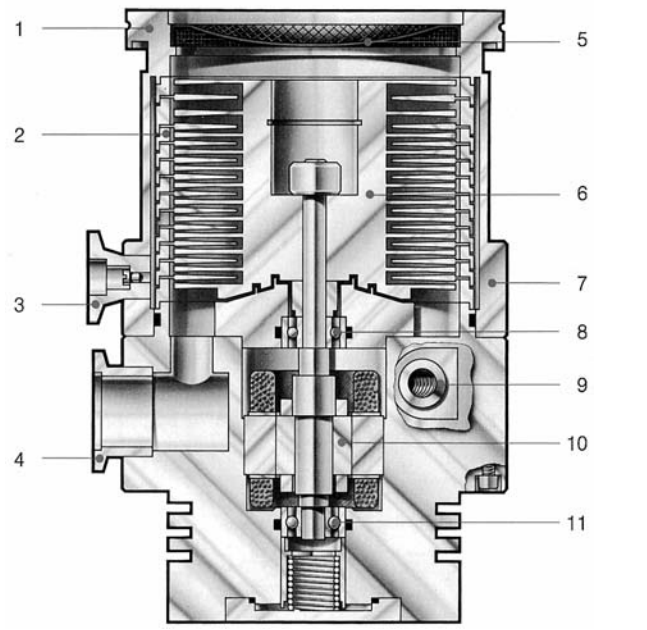
In order to meet the condition, a circumferential velocity for the rotor of the same order of magnitude as $\bar{c}$ high rotor speeds are required for turbomolecular pumps. They range from about 36,000 rpm for pumps having a large diameter rotor (TURBOVAC 1000) to 72,000 rpm in the case of smaller rotor diameters (TURBOVAC 35 / 55). Such high speeds naturally raise questions as to a reliable bearing concept. LEYBOLD offers three concepts, the advantages and disadvantages of which are detailed in the following:

- Oil lubrication / steel ball bearings
  - \+ Good compatibility with particles by circulating oil lubricant
  - \- Can only be installed vertically
  - \+ Low maintenance
- Grease lubrication / hybrid bearings
  - \+ Installation in any orientation
  - \+ Suited for mobile systems
  - ± Air cooling will do for many applications
  - \+ Lubricated for life (of the bearings)
- Free of lubricants / magnetic suspension
  - \+ No wear
  - \+ No maintenance
  - \+ Absolutely free of hydrocarbons
  - \+ Low noise and vibration levels
  - \+ Installation in any orientation

Steel ball bearings / hybrid ball bearings (ceramic ball bearings): Even a brief tear in the thin lubricating film between the balls and the races can – if the same type of material is used – result in microwelding at the points of contact. This severely reduces the service life of the bearings. By using dissimilar materials in so called hybrid bearings (races: steel, balls: ceramics) the effect of microwelding is avoided.

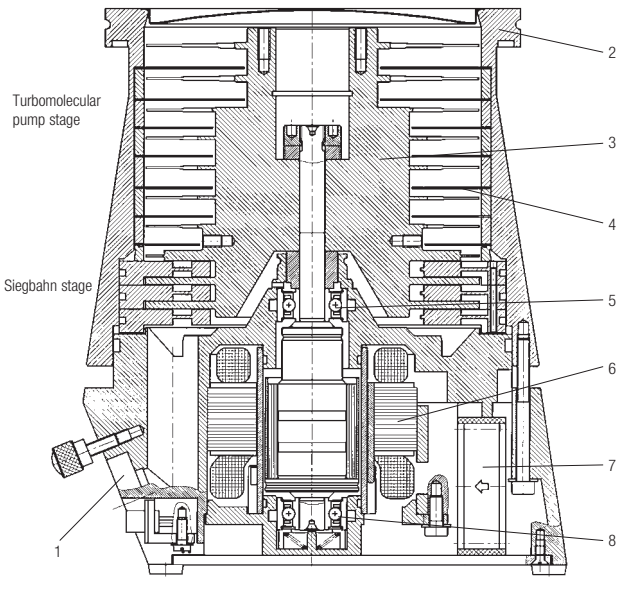
The most elegant bearing concept is that of the **magnetic suspension**. As early as 1976 LEYBOLD delivered magnetically suspended turbomolecular pumps – the legendary series 550M and 560M. At that time a purely active magnetic suspension (i.e. with electromagnets) was used. Advances in electronics and the use of permanent magnets (passive magnetic suspension) based on the "**System KFA Jülich**" permitted the magnetic suspension concept to spread widely. In this system the rotor is maintained in a stable position without contact during operation, by magnetic forces. Absolutely no lubricants are required. So-called touch down bearings are integrated for shutdown.

Fig. 2.52 shows a sectional drawing of a typical turbomolecular pump. The pump is an axial flow compressor of vertical design, the active or pumping part of which consists of a rotor (6) and a stator (2). Turbine blades are located around the circumferences of the stator and the rotor. Each rotor – stator pair of circular blade rows forms one stage, so that the assembly is composed of a multitude of stages mounted in series. The gas to be pumped arrives directly through the aperture of the inlet flange (1), that is, without any loss of conductance, at the active pumping area of the top blades of the rotor – stator assembly. This is equipped with blades of especially large radial span to allow a large annular inlet area. The gas captured by these stages is transferred to the lower compression stages, whose blades have shorter radial spans, where the gas is compressed to backing pressure or rough vacuum pressure. The turbine rotor (6) is mounted on the drive shaft, which is supported by two precision ball bearings (8 and 11), accommodated in the motor housing. The rotor shaft is directly driven by a medium-frequency motor housed in the forevacuum space within the rotor, so that no rotary shaft lead-through to the outside atmosphere is necessary. This motor is powered and automatically controlled by an external frequency converter, normally a solid-state frequency converter that ensures a very low noise level. For special applications, for example, in areas exposed to radiation, motor generator frequency converters are used.



1 High vacuum inlet flange
2 Stator pack
3 Venting flange
4 Forevacuum flange
5 Splinter guard
6 Rotor
7 Pump casing
8 Ball bearings
9 Cooling water connection
10 3-phase motor
11 Ball bearings

Fig. 2.52 Schematic diagram of a grease lubricated TURBOVAC 151 turbomolecular pump



1 Vacuum port
2 High vacuum flange
3 Rotor
4 Stator
5 Bearing
6 Motor
7 Fan
8 Bearing

Fig. 2.52a Cross section of a HY.CONE turbomolecular pump

Vacuum generation

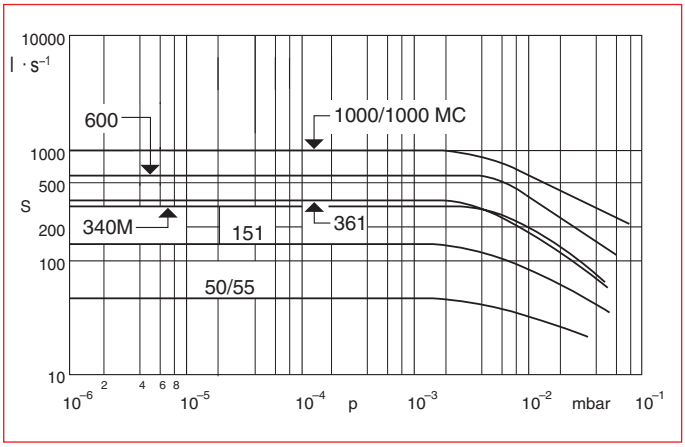


Fig. 2.53 Pumping speed for air of different turbomolecular pumps

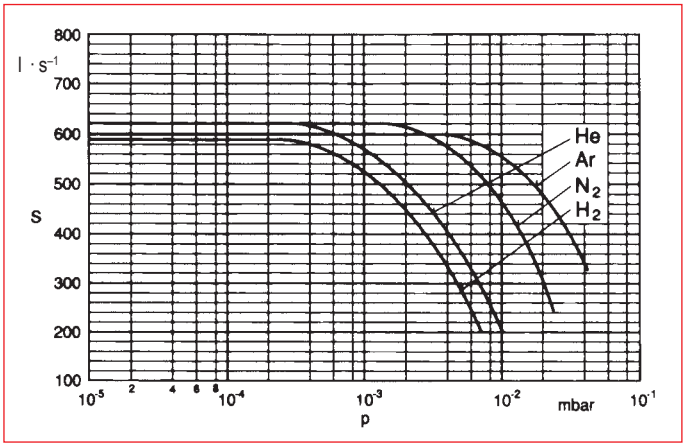


Fig. 2.54 Pumping speed curves of a TURBOVAC 600 for H_2, He, N_2 and Ar

The vertical rotor – stator configuration provides optimum flow conditions of the gas at the inlet.

To ensure vibration-free running at high rotational speeds, the turbine is dynamically balanced at two levels during its assembly.

The **pumping speed** (volume flow rate) **characteristics** of turbomolecular pumps are shown in Fig. 2.53. The pumping speed remains constant over the entire working pressure range. It decreases at intake pressures above 10^{-3} mbar, as this threshold value marks the transition from the region of molecular flow to the region of laminar viscous flow of gases. Fig. 2.54 shows also that the pumping speed depends on the type of gas.

The **compression ratio** (often also simply termed **compression**) of turbomolecular pumps is the ratio between the partial pressure of one gas component at the forevacuum flange of the pump and that at the high vacuum flange: maximum compression k_0 is to be found at zero throughput. For physical reasons, the compression ratio of turbomolecular pumps is very high for heavy molecules but considerably lower for light molecules. The relationship between compression and molecular mass is shown in Fig. 2.55. Shown in Fig. 2.56 are the compression curves of a TURBOVAC 340 M for N_2, He and H_2 as a function of the backing pressure. Because of the high compression ratio for heavy hydrocarbon molecules, turbomolecular pumps can be directly connected to a vacuum chamber without the aid of one or more cooled baffles or traps and without the risk of a measurable partial pressure for hydrocarbons in the vacuum chamber (hydrocarbon-free vacuum! – see also Fig. 2.57: residual gas spectrum above a TURBOVAC 361). As the hydrogen partial pressure

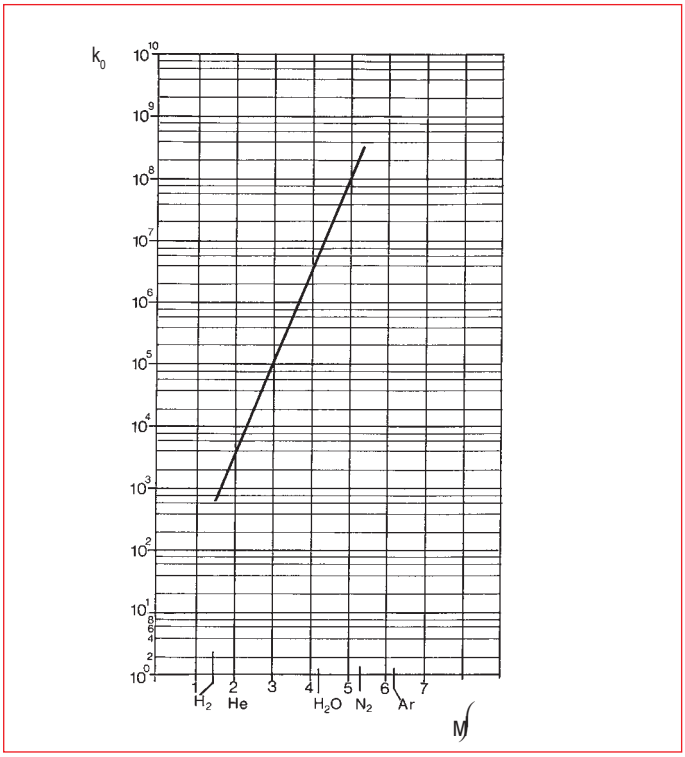


Fig. 2.55 TURBOVAC 450 – Maximum compression k_0 as a function of molar mass M

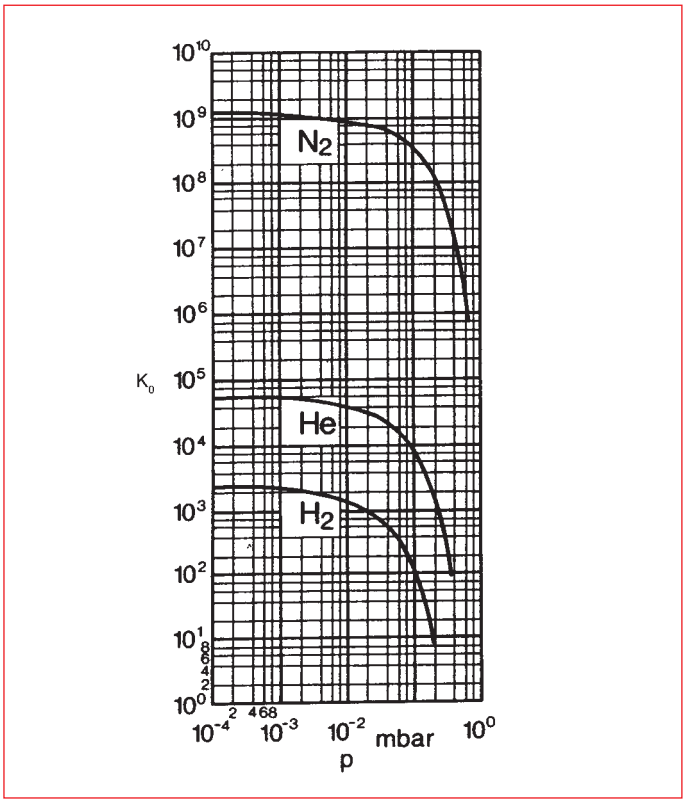


Fig. 2.56 Maximum compression k_0 of a turbomolecular pump TURBOVAC 340 M for H_2, He and N_2 as a function of backing pressure

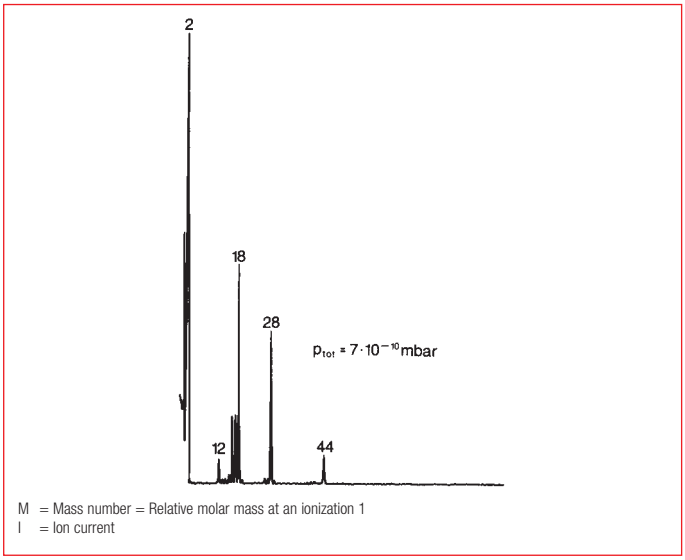


Fig. 2.57 Spectrum above a TURBOVAC 361

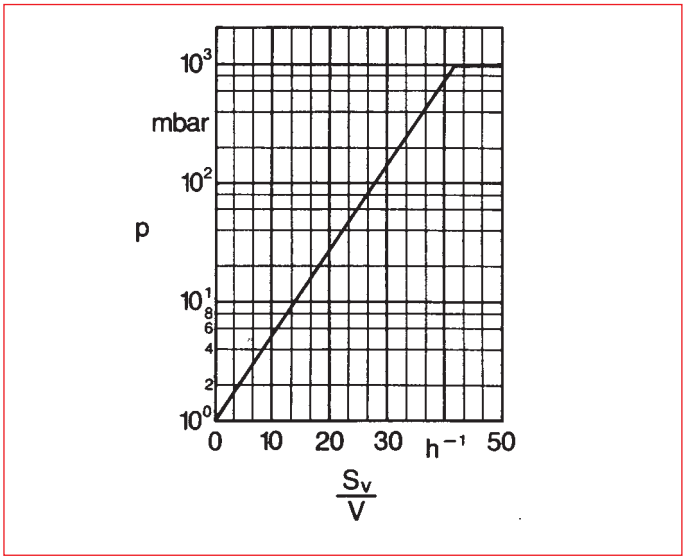


Fig. 2.58 Determination of the cut-in pressure for turbomolecular pumps when evacuating large vessels

attained by the rotary backing pump is very low, the turbomolecular pump is capable of attaining ultimate pressures in the 10^{-11} mbar range in spite of its rather moderate compression for H_2. To produce such extremely low pressures, it will, of course, be necessary to strictly observe the general rules of UHV technology: the vacuum chamber and the upper part of the turbomolecular pump must be baked out, and metal seals must be used. At very low pressures the residual gas is composed mainly of H_2 originating from the metal walls of the chamber. The spectrum in Fig. 2.57 shows the **residual gas composition** in front of the inlet of a turbomolecular pump at an ultimate pressure of $7 \cdot 10^{-10}$ mbar nitrogen equivalent. It appears that the portion of H_2 in the total quantity of gas amounts to approximately 90 to 95 % . The fraction of "heavier" molecules is considerably reduced and masses greater than 44 were not detected. An important criterion in the assessment of the quality of a residual gas spectrum are the measurable hydrocarbons from the lubricants used in the vacuum pump system. Of course an "absolutely hydrocarbon-free vacuum" can only be produced with pump systems which are free of lubricants, i.e. for example with magnetically-suspended turbomolecular pumps and dry compressing backing pumps. When operated correctly (venting at any kind of standstill) no hydrocarbons are detectable also in the spectrum of normal turbomolecular pumps.

A further development of the turbomolecular pump is the hybrid or compound turbomolecular pump. This is actually two pumps on a common shaft in a single casing. The high vacuum stage for the molecular flow region is a classic turbomolecular pump, the second pump for the viscous flow range is a molecular drag or friction pump.

LEYBOLD manufactures pumps such as the TURBOVAC 55 with an integrated Holweck stage (screw-type compressor) and, for example, the HY.CONE 60 or HY.CONE 200 with an integrated Siegbahn stage (spiral compressor). The required backing pressure then amounts to a few mbar so that the backing pump is only required to compress from about 5 to 10 mbar to atmospheric pressure. A sectional view of a HY.CONE is shown in Fig. 2.52a.

Information on the operation of turbomolecular pumps

Starting

As a rule turbomolecular pumps should generally be started together with the backing pump in order to reduce any backstreaming of oil from the backing pump into the vacuum chamber. A delayed start of the turbomolecular pump, makes sense in the case of rather small backing pump sets and large vacuum chambers. At a known pumping speed for the backing pump S_V (m^3/h) and a known volume for the vacuum chamber (m^3) it is possible to estimate the cut-in pressure for the turbomolecular pump:

Simultaneous start when

$$\frac{S_V}{V} > 40 h^{-1}$$

and delayed start when

$$\frac{S_V}{V} < 40 h^{-1}$$

at a cut-in pressure of:

$$p_{V,Start} = e^{\left(\frac{S_V}{6 \cdot V}\right)} \text{mbar} \qquad (2.24)$$

When evacuating larger volumes the cut-in pressure for turbomolecular pumps may also be determined with the aid of the diagram of Fig. 2.58.

Venting

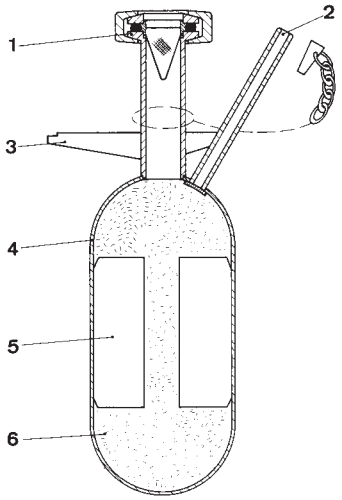
After switching off or in the event of a power failure, turbomolecular pumps should always be vented in order to prevent any backdiffusion of hydrocarbons from the forevacuum side into the vacuum chamber. After switching off the pump the cooling water supply should also be switched off to prevent the possible condensation of water vapor. In order to protect the rotor it is recommended to comply with the (minimum) venting times stated in the operating instructions. The pump should be vented (except in the case of operation with a barrier gas) via the venting flange which already contains a sintered metal throttle, so that venting may be performed using a normal valve or a power failure venting valve.

Vacuum generation

Barrier gas operation
In the case of pumps equipped with a barrier gas facility, inert gas – such as dry nitrogen – may be applied through a special flange so as to protect the motor space and the bearings against aggressive media. A special barrier gas and venting valve meters the necessary quantity of barrier gas and may also serve as a venting valve.

Decoupling of vibrations
TURBOVAC pumps are precisely balanced and may generally be connected directly to the apparatus. Only in the case of highly sensitive instruments, such as electron microscopes, is it recommended to install vibration absorbers which reduce the present vibrations to a minimum. For magnetically suspended pumps a direct connection to the vacuum apparatus will usually do because of the extremely low vibrations produced by such pumps.

For **special applications** such as operation in strong magnetic fields, radiation hazard areas or in a tritium atmosphere, please contact our Technical Sales Department which has the necessary experience and which is available to you at any time.



1 Inlet port
2 Degassing port
3 Support
4 Pump body
5 Thermal conducting vanes
6 Adsorption material (e.g. Zeolith)

Fig. 2.59 Cross section of an adsorption pump

2.1.8 Sorption pumps

The term "sorption pumps" includes all arrangements for the removal of gases and vapors from a space by sorption means. The pumped gas particles are thereby bound at the surfaces or in the interior of these agents, by either physical temperature-dependent adsorption forces (van der Waals forces), chemisorption, absorption, or by becoming embedded during the course of the continuous formation of new sorbing surfaces. By comparing their operating principles, we can distinguish between **adsorption pumps**, in which the sorption of gases takes place simply by temperature-controlled adsorption processes, and **getter pumps**, in which the sorption and retention of gases are essentially caused by the formation of chemical compounds. Gettering is the bonding of gases to pure, mostly metallic surfaces, which are not covered by oxide or carbide layers. Such surfaces always form during manufacture, installation or while venting the system. The mostly metallic highest purity getter surfaces are continuously generated either directly in the vacuum by evaporation (**evaporator pumps**) or by sputtering (**sputter pumps**) or the passivating surface layer of the getter (metal) is removed by degassing the vacuum, so that the pure material is exposed to the vacuum. This step is called activation (**NEG pumps** NEG = Non Evaporable Getter).

2.1.8.1 Adsorption pumps

Adsorption pumps (see Fig. 2.59) work according to the principle of the physical adsorption of gases at the surface of molecular sieves or other adsorption materials (e.g. activated Al_2O_3). Zeolite 13X is frequently used as an adsorption material. This alkali aluminosilicate possesses for a mass of the material an extraordinarily large surface area, about 1000 m^2/g of solid substance. Correspondingly, its ability to take up gas is considerable.

The pore diameter of zeolite 13X is about 13 Å, which is within the order of size of water vapor, oil vapor, and larger gas molecules (about 10 Å). Assuming that the mean molecular diameter is half this value, $5 \cdot 10^{-8}$ cm, about $5 \cdot 10^{18}$ molecules are adsorbed in a monolayer on a surface of 1 m^2 . For nitrogen molecules with a relative molecular mass $M_r = 28$ that corresponds to about $2 \cdot 10^{-4}$ g or 0.20 mbar · l (see also Section 1.1). Therefore, an adsorption surface of 1000 m2 is capable of adsorbing a monomolecular layer in which more than 133 mbar · l of gas is bound.

Hydrogen and light noble gases, such as helium and neon, have a relatively small particle diameter compared with the pore size of 13 Å for zeolite 13X. These gases are, therefore, very poorly adsorbed.

The adsorption of gases at surfaces is dependent not only on the temperature, but more importantly on the pressure above the adsorption surface. The dependence is represented graphically for a few gases by

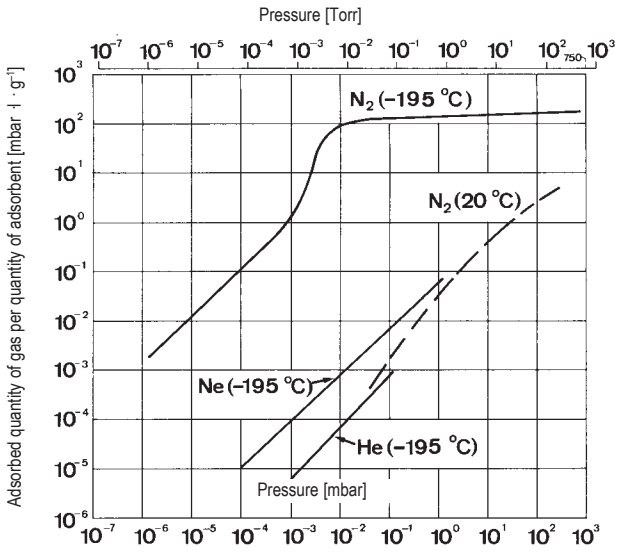


Fig. 2.60 Adsorption isotherms of zeolite 13X for nitrogen at –195 °C and 20 °C, as well as for helium and neon at –195 °C

the adsorption isotherms given in Fig. 2.60. In practice, adsorption pumps are connected through a valve to the vessel to be evacuated. It is on immersing the body of the pump in liquid nitrogen that the sorption effect is made technically useful. Because of the different adsorption properties, the pumping speed and ultimate pressure of an adsorption pump are different for the various gas molecules: the best values are achieved for nitrogen, carbon dioxide, water vapor, and hydrocarbon vapors. Light noble gases are hardly pumped at all because the diameter of the particles is small compared to the pores of the zeolite. As the sorption effect decreases with increased coverage of the zeolite surfaces, the pumping speed falls off with an increasing number of the particles already adsorbed. The pumping speed of an adsorption pump is, therefore, dependent on the quantity of gas already pumped and so is not constant with time.

The ultimate pressure attainable with adsorption pumps is determined in the first instance by those gases that prevail in the vessel at the beginning of the pumping process and are poorly or not at all adsorbed (e.g. neon or helium) at the zeolite surface. In atmospheric air, a few parts per million of these gases are present. Therefore, pressures < 10^{-2} mbar can be obtained.

If pressures below 10^{-3} mbar exclusively are to be produced with adsorption pumps, as far as possible no neon or helium should be present in the gas mixture.

After a pumping process, the pump must be warmed only to room temperature for the adsorbed gases to be given off and the zeolite is regenerated for reuse. If air (or damp gas) containing a great deal of water vapor has been pumped, it is recommended to bake out the pump completely dry for a few hours at 200 °C or above.

To pump out larger vessels, several adsorption pumps are used in parallel or in series. First, the pressure is reduced from atmospheric pressure to a few millibars by the first stage in order to “capture” many noble gas molecules of helium and neon. After the pumps of this stage have been saturated, the valves to these pumps are closed and a previously closed valve to a further adsorption pump still containing clean adsorbent is opened so that this pump may pump down the vacuum chamber to the next lower pressure level. This procedure can be continued until the ultimate pressure cannot be further improved by adding further clean adsorption pumps.

2.1.8.2 Sublimation pumps

Sublimation pumps are sorption pumps in which a getter material is evaporated and deposited on a cold inner wall as a getter film. On the surface of such a getter film the gas molecules form stable compounds, which have an immeasurably low vapor pressure. The active getter film is renewed by subsequent evaporations. Generally titanium is used in sublimation pumps as the getter. The titanium is evaporated from a wire made of a special alloy of a high titanium content which is heated by an electric current. Although the optimum sorption capacity (about one nitrogen atom for each evaporated titanium atom) can scarcely be obtained in practice, titanium sublimation pumps have an extraordinarily high pumping speed for active gases, which, particularly on starting processes or on the sudden evolution of greater quantities of gas, can be rapidly pumped away. As sublimation pumps function as auxiliary pumps (boosters) to sputter-ion pumps and turbomolecular pumps, their installation is often indispensable (like the “boosters” in vapor ejector pumps; see Section 2.1.6.2).

2.1.8.3 Sputter-ion pumps

The pumping action of sputter-ion pumps is based on sorption processes that are initiated by ionized gas particles in a Penning discharge (cold cathode discharge). By means of “paralleling many individual Penning cells” the sputter ion pump attains a sufficiently high pumping speed for the individual gases.

Operation of sputter-ion pumps

The ions impinge upon the cathode of the cold cathode discharge electrode system and sputter the cathode material (titanium). The titanium deposited at other locations acts as a getter film and adsorbs reactive gas particles (e.g., nitrogen, oxygen, hydrogen). The energy of the ionized gas particles is not only high enough to sputter the cathode material but also to let the impinging ions penetrate deeply into the cathode material (ion implantation). This sorption process “pumps” ions of all types, including ions of gases which do not chemically react with the sputtered titanium film, i.e. mainly noble gases.

The following arrangement is used to produce the ions: stainless-steel, cylindrical anodes are closely arranged between, with their axes perpendicular to, two parallel cathodes (see Fig. 2.61). The cathodes are at negative potential (a few kilovolts) against the anode. The entire electrode system is maintained in a strong, homogeneous magnetic field of a flux density of B = 0.1 T, (T = Tesla = 10^4 Gauss) produced by a permanent magnet attached to the outside of the pump’s casing. The gas discharge produced by the high tension contains electrons and ions. Under the influence of the magnetic field the electrons travel along long spiral tracks (see Fig. 2.61) until they impinge on the anode cylinder of the corresponding cell. The long track increases ion yield, which even at low gas densities (pressures) is sufficient to maintain a self-sustained gas discharge. A supply of electrons from a hot cathode is not required. Because of their great mass, the movement of the ions is unaffected by the magnetic field of the given order of magnitude; they flow off along the shortest path and bombard the cathode.

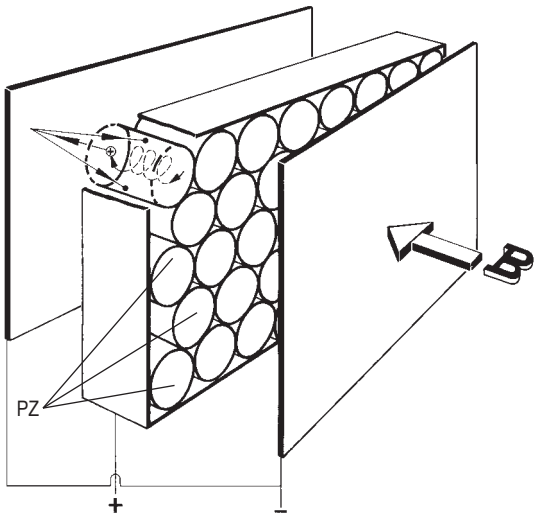


Fig. 2.61 Operating principle of a sputter-ion pump

Vacuum generation

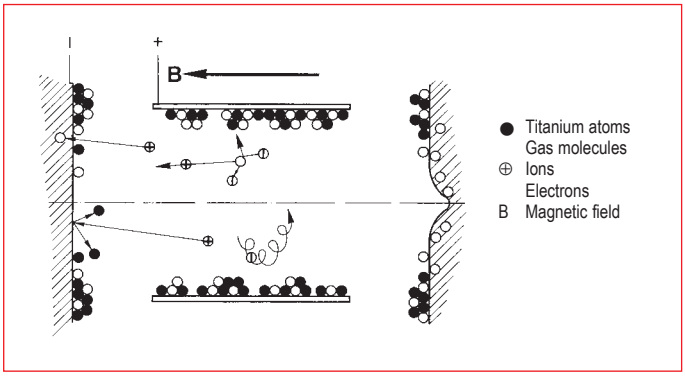


Fig. 2.62 Electrode configuration in a diode sputter-ion pump

The discharge current i is proportional to the number density of neutral particles n_0, the electron density n^-, and the length l of the total discharge path:

$$i = n_0 \cdot n^- \cdot \sigma \cdot l \qquad (2.25)$$

The effective cross section s for ionizing collisions depends on the type of gas. According to (2.25), the discharge current i is a function of the number particle density n_0, as in a Penning gauge, and it can be used as a measure of the pressure in the range from 10^{-4} to 10^{-8} mbar. At lower pressures the measurements are not reproducible due to interferences from field emission effects.

In **diode-type, sputter-ion pumps**, with an electrode system configuration as shown in Fig. 2.62, the getter films are formed on the anode surfaces and between the sputtering regions of the opposite cathode. The ions are buried in the cathode surfaces. As cathode sputtering proceeds, the buried gas particles are set free again. Therefore, the pumping action for noble gases that can be pumped only by ion burial will vanish after some time and a "memory effect" will occur.

Unlike diode-type pumps, **triode sputter-ion pumps** exhibit excellent stability in their pumping speed for noble gases because sputtering and film forming surfaces are separated. Fig. 2.63 shows the electrode configuration of triode sputter-ion pumps. Their greater efficiency for pumping noble gases is explained as follows: the geometry of the system favors grazing incidence of the ions on the titanium bars of the cathode grid, whereby the sputtering rate is considerably higher than with perpendicular incidence. The sputtered titanium moves in about the same direction as the incident ions. The getter films form preferentially on the third electrode, the target

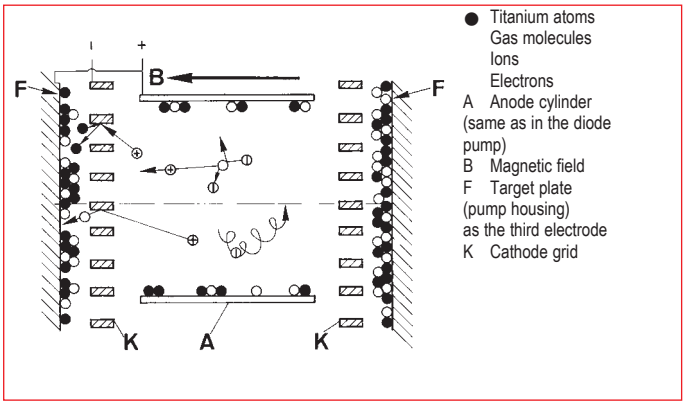


Fig. 2.63 Electrode configuration in a triode sputter-ion pump

plate, which is the actual wall of the pump housing. There is an increasing yield of ionized particles that are grazingly incident on the cathode grid where they are neutralized and reflected and from which they travel to the target plate at an energy still considerably higher than the thermal energy $1/2 \cdot k \cdot T$ of the gas particles. The energetic neutral particles can penetrate into the target surface layer, but their sputtering effect is only negligible. These buried or implanted particles are finally covered by fresh titanium layers. As the target is at positive potential, any positive ions arriving there are repelled and cannot sputter the target layers. Hence the buried noble gas atoms are not set free again. **The pumping speed of triode sputter-ion pumps for noble gases does not decrease during the operation of the pump.**

The pumping speed of sputter-ion pumps depends on the pressure and the type of gas. It is measured according to the methods stated in DIN 28 429 and PNEUROP 5615. The pumping speed curve S(p) has a maximum. The nominal pumping speed S_n is given by the maximum of the pumping speed curve for air whereby the corresponding pressure must be stated.

For air, nitrogen, carbon dioxide and water vapor, the pumping speed is practically the same. Compared with the pumping speed for air, the pumping speeds of sputter-ion pumps for other gases amount to approximately:

Hydrogen	150 to 200 %
Methane	100 %
Other light hydrocarbons	80 to 120 %
Oxygen	80 %
Argon	30 %
Helium	28 %

Sputter-ion pumps of the triode type excel in contrast to the diode-type pumps in high-noble gas stability. Argon is pumped stably even at an inlet pressure of $1 \cdot 10^{-5}$ mbar. The pumps can be started without difficulties at pressures higher than $1 \cdot 10^{-2}$ mbar and can operate continuously at an air inlet producing a constant air pressure of $5 \cdot 10^{-5}$ mbar. A new kind of design for the electrodes extends the service life of the cathodes by 50 %.

Influence on processes in the vacuum chamber by magnetic stray fields and stray ions from the sputter-ion pump.
The high-magnetic-field strength required for the pumping action leads inevitably to stray magnetic fields in the neighborhood of the magnets. As a result, processes in the vacuum chamber can be disturbed in some cases, so the sputter-ion pump concerned should be provided with a screening arrangement. The forms and kinds of such a screening arrangement can be regarded as at an optimum if the processes taking place in the vacuum chamber are disturbed by no more than the earth's magnetic field which is present in any case.

Fig. 2.64 shows the magnetic stray field at the plane of the intake flange of a sputter-ion pump IZ 270 and also at a parallel plane 150 mm above. If stray ions from the discharge region are to be prevented from reaching the vacuum chamber, a suitable screen can be set up by a metal sieve at opposite potential in the inlet opening of the sputter-ion pump (ion barrier). This, however, reduces the pumping speed of the sputter-ion pump depending on the mesh size of the selected metal sieve.

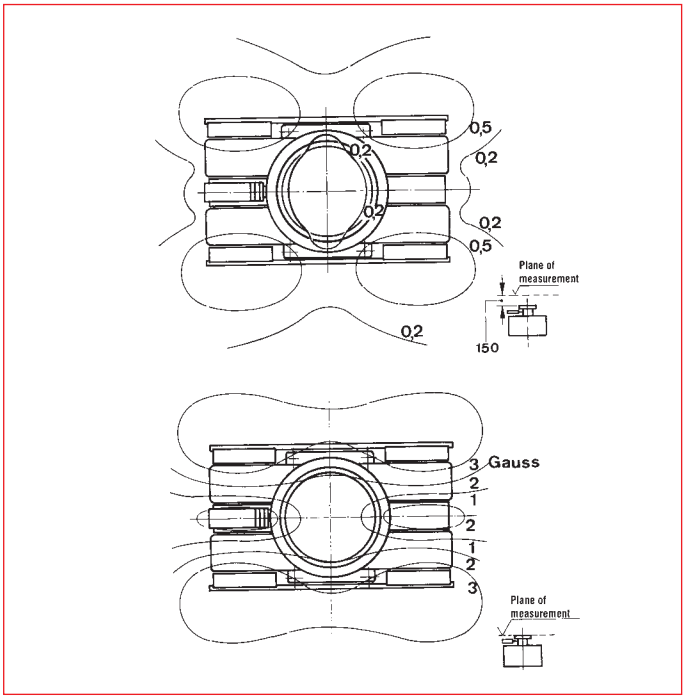


Fig. 2.64 Stray magnetic field of a sputter-ion pump in two places parallel to the inlet flange (inserts) curves show lines of constant magnetic induction B in Gauss.1 Gauss = $1 \cdot 10^{-4}$ Tesla

2.1.8.4 Non evaporable getter pumps (NEG Pumps)

The non evaporable getter pump operates with a non evaporable, compact getter material, the structure of which is porous at the atomic level so that it can take up large quantities of gas. The gas molecules adsorbed on the surface of the getter material diffuse rapidly inside the material thereby making place for further gas molecules impinging on the surface. The non evaporable getter pump contains a heating element which is used to heat the getter material to an optimum temperature depending on the type of gas which is preferably to be pumped. At a higher temperature the getter material which has been saturated with the gas is regenerated (activated). As the getter material, mostly zirconium-aluminum alloys are used in the form of strips. The special properties of NEG pumps are:

- constant pumping speed in the HV and UHV ranges
- no pressure restrictions up to about 12 mbar
- particularly high pumping speed for hydrogen and its isotopes
- after activation the pump can often operate at room temperature and will then need no electrical energy
- no interference by magnetic fields
- hydrocarbon-free vacuum
- free of vibrations
- low weight

NEG pumps are mostly used in combination with other UHV pumps (turbomolecular and cryopumps). Such combinations are especially useful when wanting to further reduce the ultimate pressure of UHV systems, since hydrogen contributes mostly to the ultimate pressure in an UHV system, and for which NEG pumps have a particularly high pumping speed, whereas the pumping effect for H_2 of other pumps is low. Some typical examples for applications in which NEG pumps are used are particle accelerators and similar research systems, surface analysis instruments, SEM columns and sputtering systems. NEG pumps are manufactured offering pumping speeds of several `/s to about 1000 l/s. Custom pumps are capable of attaining a pumping speed for hydrogen which is by several orders of magnitude higher.

2.1.9 Cryopumps

As you may have observed water condenses on cold water mains or windows and ice forms on the evaporator unit in your refrigerator. This effect of condensation of gases and vapors on cold surfaces, water vapor in particular, as it is known in every day life, occurs not only at atmospheric pressure but also in vacuum.

This effect has been utilized for a long time in condensers (see 2.1.5) mainly in connection with chemical processes; previously the baffle on diffusion pumps used to be cooled with refrigerating machines. Also in a sealed space (vacuum chamber) the formation of condensate on a cold surface means that a large number of gas molecules are removed from the volume: they remain located on the cold surface and do not take part any longer in the hectic gas atmosphere within the vacuum chamber. We then say that the particles have been pumped and talk of cryopumps when the "pumping effect" is attained by means of cold surfaces.

Cryo engineering differs from refrigeration engineering in that the temperatures involved in cryo engineering are in the range below 120 K (< -153 °C). Here we are dealing with two questions:

a) What cooling principle is used in cryo engineering or in cryopumps and how is the thermal load of the cold surface lead away or reduced?

b) What are the operating principles of the cryopumps?

2.1.9.1 Types of cryopump

Depending on the cooling principle a difference is made between

- Bath cryostats
- Continuous flow cryopumps
- Refrigerator cryopumps

In the case of **bath cryostats** – in the most simple case a cold trap filled with LN_2 (liquid nitrogen) – the pumping surface is cooled by direct contact with a liquefied gas. On a surface cooled with LN_2 (T ≈ 77 K) H_2O and CO_2 are able to condense. On a surface cooled to ≈ 10 K all gases except He and Ne may be pumped by way of condensation. A surface cooled with liquid helium (T ≈ 4.2 K) is capable of condensing all gases except helium.

In **continuous flow cryopumps** the cold surface is designed to operate as a heat exchanger. Liquid helium in sufficient quantity is pumped by an auxiliary pump from a reservoir into the evaporator in order to attain a sufficiently low temperature at the cold surface (cryopanel).

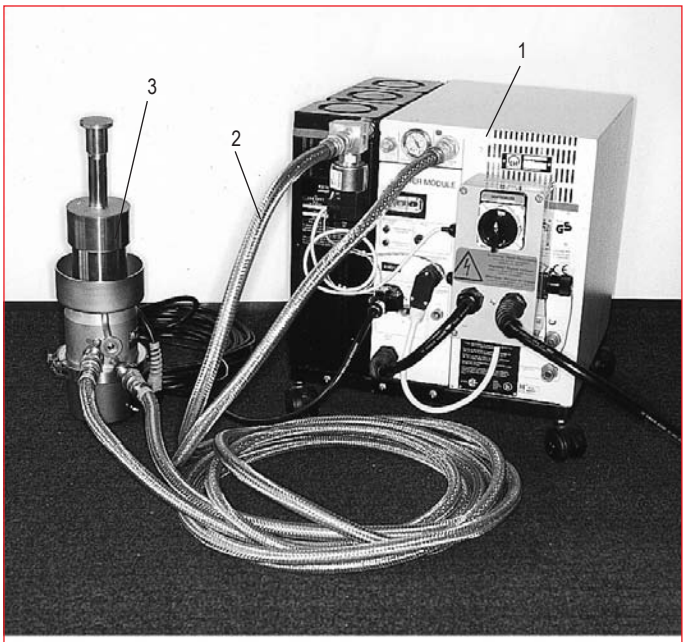
The liquid helium evaporates in the heat exchanger and thus cools down the cryopanel. The waste gas which is generated (He) is used in a second heat exchanger to cool the baffle of a thermal radiation shield which protects the system from thermal radiation coming from the outside. The cold helium exhaust gas ejected by the helium pump is supplied to a helium recovery unit. The temperature at the cryopanels can be controlled by controlling the helium flow.

Today **refrigerator cryopumps** are being used almost exclusively (cold upon demand). These pumps operate basically much in the same way as a common household refrigerator, whereby the following thermodynamic cycles using helium as the refrigerant may be employed:

- Gifford-McMahon process
- Stirling process
- Brayton process
- Claude process

The Gifford-McMahon process is mostly used today and this process is that which has been developed furthest. It offers the possibility of separating the locations for the large compressor unit and the expansion unit in which the refrigeration process takes place. Thus a compact and low vibration cold source can be designed. The cryopumps series-manufactured by LEYBOLD operate with two-stage cold heads according to the Gifford-McMahon process which is discussed in detail in the following.

The entire scope of a refrigerator cryopump is shown in Fig. 2.65 and consists of the compressor unit (1) which is linked via flexible pressure lines (2) – and thus vibration-free – to the cryopump (3). The cryopump itself consists of the pump casing and the cold head within. Helium is used as the refrigerant which circulates in a closed cycle with the aid of the compressor.



1 Compressor unit
2 Flexible pressure lines
3 Cold head (without condensation surfaces)

Fig. 2.65 All items of a refrigerator cryopump

2.1.9.2 The cold head and its operating principle (Fig. 2.66)

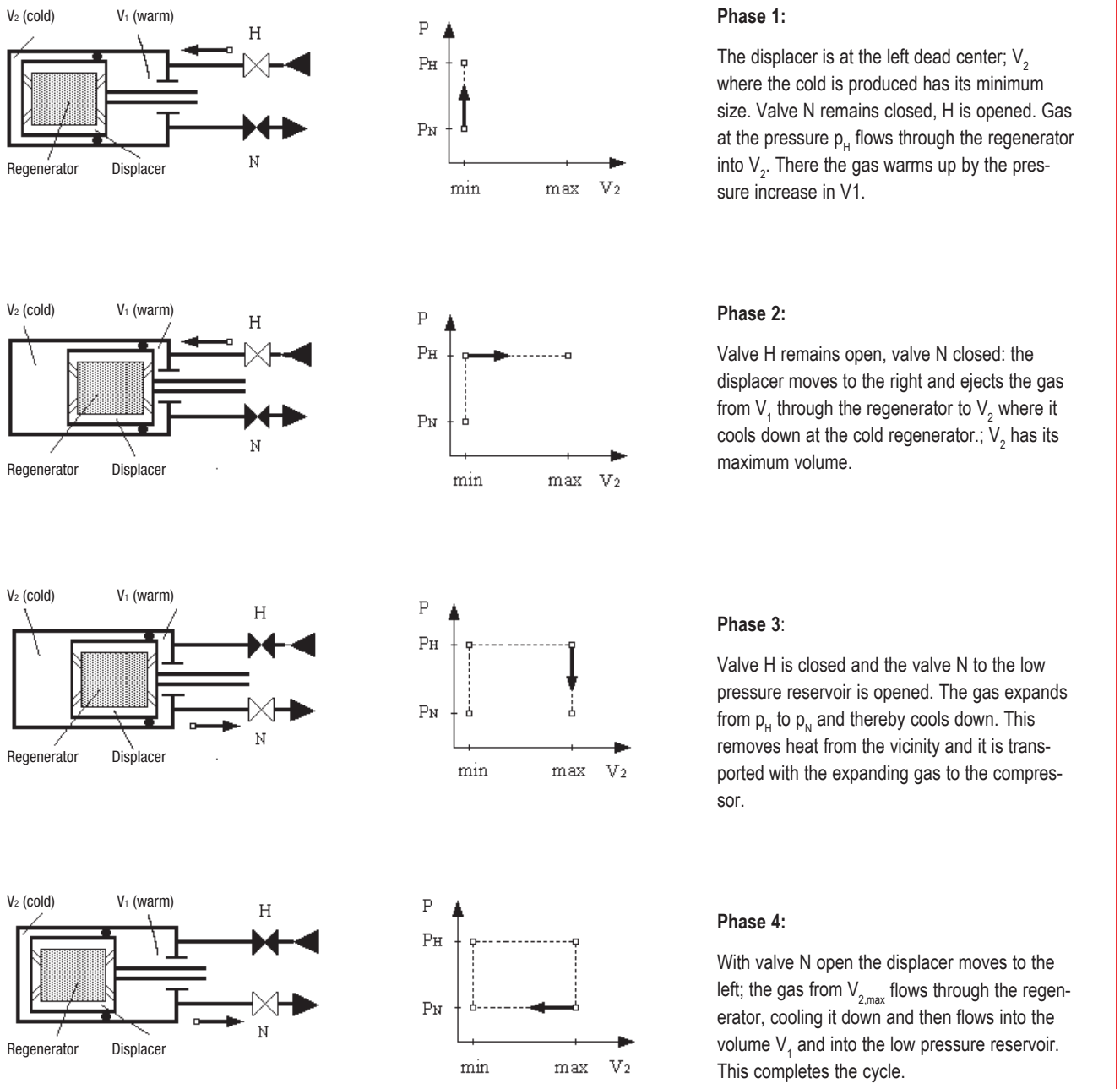
Within the cold head, a cylinder is divided into two working spaces V_1 and V_2 by a displacer. During operation the right space V_1 is warm and the left space V_2 is cold. At a displacer frequency f the refrigerating power W of the refrigerator is:

$$W = (V_{2,max} - V_{2,min}) \cdot (p_H - p_N) \cdot f \qquad (2.26)$$

The displacer is moved to and fro pneumatically so that the gas is forced through the displacer and thus through the regenerator located inside the displacer. The regenerator is a heat accumulator having a large heat exchanging surface and capacity, which operates as a heat exchanger within the cycle. Outlined in Fig. 2.66 are the four phases of refrigeration in a single-stage refrigerator cold head operating according to the Gifford-McMahon principle.

The two-stage cold head

The series-manufactured refrigerator cryopumps from LEYBOLD use a two-stage cold head operating according to the Gifford-McMahon principle (see Fig. 2.67). In two series connected stages the temperature of the helium is reduced to about 30 K in the first stage and further to about 10 K in the second stage. The attainable low temperatures depend among other



Phase 1:

The displacer is at the left dead center; V_2 where the cold is produced has its minimum size. Valve N remains closed, H is opened. Gas at the pressure p_H flows through the regenerator into V_2. There the gas warms up by the pressure increase in V1.

Phase 2:

Valve H remains open, valve N closed: the displacer moves to the right and ejects the gas from V_1 through the regenerator to V_2 where it cools down at the cold regenerator.; V_2 has its maximum volume.

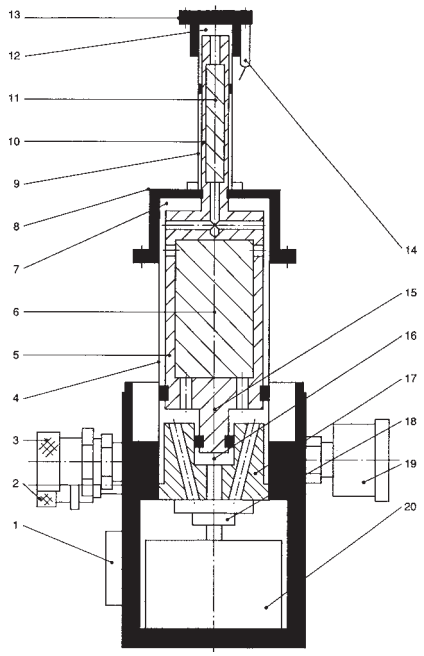
Phase 3:

Valve H is closed and the valve N to the low pressure reservoir is opened. The gas expands from p_H to p_N and thereby cools down. This removes heat from the vicinity and it is transported with the expanding gas to the compressor.

Phase 4:

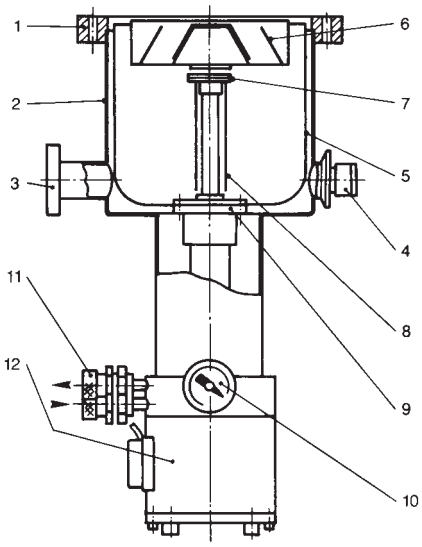
With valve N open the displacer moves to the left; the gas from $V_{2,max}$ flows through the regenerator, cooling it down and then flows into the volume V_1 and into the low pressure reservoir. This completes the cycle.

Fig. 2.66 Refrigerating phases using a single-stage cold head operating according to the Gifford-McMahon process



1 Electric connections and current feedthrough for the motor in the cold head
2 He high pressure connection
3 He low pressure connection
4 Cylinder, 1st stage
5 Displacer, 1st stage
6 Regenerator, 1st stage
7 Expansion volume, 1st stage
8 1st (cooling) stage (copper flange)
9 Cylinder, 2nd stage
10 Displacer, 2nd stage
11 Regenerator, 2nd stage
12 Expansion volume, 2nd stage
13 2nd (cooling) stage (copper flange)
14 Measurement chamber for the vapor pressure
15 Control piston
16 Control volume
17 Control disk
18 Control valve
19 Gauge for the hydrogen vapor pressure thermometer
20 Motor in the cold head

Fig. 2.67 Two-stage cold head



1 High vacuum flange
2 Pump casing
3 Forevacuum flange
4 Safety valve for gas discharge
5 Thermal radiation shield
6 Baffle
7 2nd stage of the cold head (≈10 K);
8 Cryopanels
9 1st stage of the cold head (≈ 50–80 K)
10 Gauge for the hydrogen vapor pressure thermometer
11 Helium gas connections
12 Motor of the cold head with casing and electric connections

Fig. 2.68 Design of a refrigerator cryopump (schematic)

things on the type of regenerator. Commonly copperbronze is used in the regenerator of the first stage and lead in the second stage. Other materials are available as regenerators for special applications like cryostats for extremely low temperatures (T < 10 K). The design of a two-stage cold head is shown schematically in Fig. 2.67. By means of a control mechanism with a motor driven control valve (18) with control disk (17) and control holes first the pressure in the control volume (16) is changed which causes the displacers (6) of the first stage and the second stage (11) to move; immediately thereafter the pressure in the entire volume of the cylinder is equalized by the control mechanism. The cold head is linked via flexible pressure lines to the compressor.

2.1.9.3 The refrigerator cryopump

Fig. 2.68 shows the design of a cryopump. It is cooled by a two-stage cold head. The thermal radiation shield (5) with the baffle (6) is closely linked thermally to the first stage (9) of the cold head. For pressures below 10^{-3} mbar the thermal load is caused mostly by thermal radiation. For this reason the second stage (7) with the condensation and cryosorption panels (8) is surrounded by the thermal radiation shield (5) which is black on the inside and polished as well as nickel plated on the outside. Under no-load conditions the baffle and the thermal radiation shield (first stage) attain a temperature ranging between 50 to 80 K at the cryopanels and about 10 K at the second stage. The surface temperatures of these cryopanels are decisive to the actual pumping process. These surface temperatures depend on the refrigerating power supplied by the cold head, and the thermal conduction properties in the direction of the pump's casing. During operation of the cryopump, loading caused by the gas and the heat of condensation results in further warming of the cryopanels. The surface temperature does not only depend on the temperature of the cryopanel, but also on the temperature of the gas which has already been frozen on to the cryopanel. The cryopanels (8) attached to the second stage (7) of the cold head are coated with activated charcoal on the inside in order to be able to pump gases which do not easily condense and which can only be pumped by cryosorption (see 2.1.9.4).

2.1.9.4 Bonding of gases to cold surfaces

The thermal conductivity of the condensed (solid) gases depends very much on their structure and thus on the way in which the condensate is produced. Variations in thermal conductivity over several orders of magnitude are possible! As the condensate increases in thickness, thermal resistance and thus the surface temperature increase subsequently reducing the pumping speed. The maximum pumping speed of a newly regenerated pump is stated as its nominal pumping speed. The bonding process for the various gases in the cryopump is performed in three steps: first the mixture of different gases and vapors meets the baffle which is at

a temperature of about 80 K. Here mostly H_2O and CO_2 are condensed. The remaining gases penetrate the baffle and impinge in the outside of the cryopanel of the second stage which is cooled to about 10 K. Here gases like N_2, O_2 or Ar will condense. Only H_2, He and Ne will remain. These gases can not be pumped by the cryopanels and these pass after several impacts with the thermal radiation shield to the inside of these panels which are coated with an adsorbent (cryosorption panels) where they are bonded by cryosorption. Thus for the purpose of considering a cryopump the gases are divided into three groups depending at which temperatures within the cryopump their partial pressure drops below 10^{-9} mbar:

1st group:
$p_s < 10^{-9}$ mbar at T ≈ 77K (LN_2): H_2O, CO_2
2nd group:
$p_s < 10^{-9}$ mbar at T ≈ 20K: N_2, O_2, Ar
3rd group:
$p_s < 10^{-9}$ mbar at T < 4.2K: H_2, He, Ne

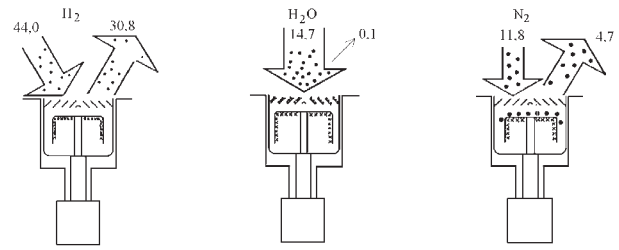
A difference is made between the different bonding mechanisms as follows:

Cryocondensation is the physical and reversible bonding of gas molecules through Van der Waals forces on sufficiently cold surfaces of the same material. The bond energy is equal to the energy of vaporization of the solid gas bonded to the surface and thus decreases as the thickness of the condensate increases as does the vapor pressure. **Cryosorption** is the physical and reversible bonding of gas molecules through Van der Waals forces on sufficiently cold surfaces of other materials. The bond energy is equal to the heat of adsorption which is greater than the heat of vaporization. As soon as a monolayer has been formed, the following molecules impinge on a surface of the same kind (sorbent) and the process transforms into cryocondensation. The higher bond energy for cryocondensation prevents the further growth of the condensate layer thereby restricting the capacity for the adsorbed gases. However, the adsorbents used, like activated charcoal, silica gel, alumina gel and molecular sieve, have a porous structure with very large specific surface areas of about 10^6 m^2/kg. **Cryotrapping** is understood as the inclusion of a low boiling point gas which is difficult to pump such as hydrogen, in the matrix of a gas having a higher boiling point and which can be pumped easily such as Ar, CH_4 or CO_2. At the same temperature the condensate mixture has a saturation vapor pressure which is by several orders of magnitude lower than the pure condensate of the gas with the lower boiling point.

2.1.9.5 Pumping speed and position of the cryopanels

Considering the position of the cryopanels in the cryopump, the conductance from the vacuum flange to this surface and also the subtractive pumping sequence (what has already condensed at the baffle can not arrive at the second stage and consume capacity there), the situation arises as shown in Fig. 2.69.

The gas molecules entering the pump produce the area related theoretical pumping speed according the equation 2.29a with T = 293 K. The different pumping speeds have been combined for three representative gases H_2, N_2 and H_20 taken from each of the aforementioned groups. Since water vapor is pumped on the entire entry area of the cryopump, the pumping speed



	Hydrogen	Water vapor	Nitrogen
Area related conductance of the intake flange in l / s · cm²:	43.9	14.7	11.8
Area related pumping speed of the cryopump in l / s · cm²:	13.2	14.6	7.1
Ratio between pumping speed and conductance:	30 %	99 %	60 %

Fig. 2.69 Cryopanels – temperature and position define the efficiency in the cryopump

measured for water vapor corresponds almost exactly to the theoretical pumping speed calculated for the intake flange of the cryopump. N_2 on the other hand must first overcome the baffle before it can be bonded on to the cryocondensation panel. Depending on the design of the baffle, 30 to 50 percent of all N_2 molecules are reflected.

H_2 arrives at the cryosorption panels after further collisions and thus cooling of the gas. In the case of optimally designed cryopanels and a good contact with the active charcoal up to 50 percent of the H_2 which has overcome the baffle can be bonded. Due to the restrictions regarding access to the pumping surfaces and cooling of the gas by collisions with the walls inside the pump before the gas reaches the pumping surface, the measured pumping speed for these two gases amounts only to a fraction of the theoretical pumping speed. The part which is not pumped is reflected chiefly by the baffle. Moreover, the adsorption probability for H_2 differs between the various adsorbents and is < 1, whereas the probabilities for the condensation of water vapor and N_2 ≈ 1.

Three differing capacities of a pump for the gases which can be pumped result from the size of the three surfaces (baffle, condensation surface at the outside of the second stage and sorption surface at the inside of the second stage). In the design of a cryopump, a mean gas composition (air) is assumed which naturally does not apply to all vacuum processes (sputtering processes, for example. See 2.1.9.6 “Partial Regeneration”).

2.1.9.6 Characteristic quantities of a cryopump

The characteristic quantities of a cryopump are as follows (in no particular order):

- Cooldown time
- Crossover value
- Ultimate pressure
- Capacity
- Refrigerating power and net refrigerating power
- Regeneration time
- Throughput and maximum pV flow
- Pumping speed
- Service life / duration of operation
- Starting pressure

Vacuum generation

Cooldown time: The cooldown time of cryopumps is the time span from start-up until the pumping effect sets in. In the case of refrigerator cryopumps the cooldown time is stated as the time it takes for the second stage of the cold head to cool down from 293 K to 20 K.

Crossover value: The crossover value is a characteristic quantity of an already cold refrigerator cryopump. It is of significance when the pump is connected to a vacuum chamber via an HV / UHV valve. The crossover value is that quantity of gas with respect to T_n=293 K which the vacuum chamber may maximally contain so that the temperature of the cryopanels does not increase above 20 K due to the gas burst when opening the valve. The crossover value is usually stated as a pV value in in mbar · l.

The crossover value and the chamber volume V result in the crossover pressure p_c to which the vacuum chamber must be evacuated first before opening the valve leading to the cryopump. The following may serve as a guide:

$$p_c \leq \frac{35}{V} \cdot \dot{Q}_2(20K)\ \text{mbar} \qquad (2.27)$$

V = Volume of the vacuum chamber (l),

$\dot{Q}_2$(20K) = Net refrigerating capacity in Watts, available at the second stage of the cold head at 20 K.

Ultimate pressure p_{end}: For the case of cryocondensation (see Section 2.1.9.4) the ultimate pressure can be calculated by:

$$p_{end} = p_S(T_K) \cdot \sqrt{\frac{T_G}{T_K}} \qquad (2.28)$$

p_S is the saturation vapor pressure of the gas or gases which are to be pumped at the temperature T_K of the cryopanel and T_G is the gas temperature (wall temperature in the vicinity of the cryopanel).

Example: With the aid of the vapor pressure curves in Fig. 9.15 for H_2 and N_2 the ultimate pressures summarized in Table 2.6 at T_G = 300 K result.

The Table shows that for hydrogen at temperatures T < 3 K at a gas

T_K (K)	Ultimate pressure (according to equ. 2.28)	Ult. press. (mbar) H_2	Ult. press.(mbar) N_2
2.5	$10.95 \cdot p_s$	$3.28 \cdot 10^{-14}$	immeasurably low
4.2	$8.66 \cdot p_s$	$4.33 \cdot 10^{-9}$	immeasurably low
20	$3.87 \cdot p_s$	$3.87 \cdot 10^{+3}$	$3.87 \cdot 10^{-11}$

Table 2.6 Ultimate temperatures at a wall temperature of 300 K

temperature of T_G= 300 K (i.e. when the cryopanel is exposed to the thermal radiation of the wall) sufficiently low ultimate pressures can be attained. Due to a number of interfering factors like desorption from the wall and leaks, the theoretical ultimate pressures are not attained in practice.

Capacity C (mbar · l): The capacity of a cryopump for a certain gas is that quantity of gas (p_V value at T_n = 293 K) which can be bonded by the cryopanels before the pumping speed for this type of gas G drops to below 50 % of its initial value.

The capacity for gases which are pumped by means of cryosorption depends on the quantity and properties of the sorption agent; it is pressure dependent and generally by several orders of magnitude lower compared to the pressure independent capacity for gases which are pumped by means of cryocondensation.

Refrigerating power $\dot{Q}$ (W): The refrigerating power of a refrigeration source at a temperature T gives the amount of heat that can be extracted by the refrigerating source whilst still maintaining this temperature. In the case of refrigerators it has been agreed to state for single-stage cold heads the refrigerating power at 80 K and for two-stage cold heads the refrigerating power for the first stage at 80 K and for the second stage at 20 K when simultaneously loading both stages thermally. During the measurement of refrigerating power the thermal load is generated by electric heaters. The refrigerating power is greatest at room temperature and is lowest (Zero) at ultimate temperature.

Net refrigerating power $\dot{Q}$ (W): In the case of refrigerator cryopumps the net refrigerating power available at the usual operating temperatures (T1 < 80 K, T2 < 20 K) substantially defines the throughput and the crossover value. The net. refrigerating power is – depending on the configuration of the pump – much lower than the refrigerating power of the cold head used without the pump.

pV flow see 1.1

Regeneration time: As a gas trapping device, the cryopump must be regenerated after a certain period of operation. Regeneration involves the removal of condensed and adsorbed gases from the cryopanels by heating. The regeneration can be run fully or only partially and mainly differs by the way in which the cryopanels are heated.

In the case of **total regeneration** a difference is made between:

1. Natural warming: after switching off the compressor, the cryopanels at first warm up only very slowly by thermal conduction and then in addition through the released gases.
2. Purge gas method: the cryopump is warmed up by admitting warm purging gas.
3. Electric heaters: the cryopanels of the cryopump are warmed up by heaters at the first and second stages. The released gases are discharged either through an overpressure valve (purge gas method) or by mechanical backing pumps. Depending on the size of the pump one will have to expect a regeneration time of several hours.

Partial regeneration: Since the limitation in the service life of a cryopump depends in most applications on the capacity limit for the gases nitrogen, argon and hydrogen pumped by the second stage, it will often be required to regenerate only this stage. Water vapor is retained during partial regeneration by the baffle. For this, the temperature of the first stage must be maintained below 140 K or otherwise the partial pressure of the water vapor would become so high that water molecules would contaminate the adsorbent on the second stage.

In 1992, LEYBOLD was the first manufacturer of cryopumps to develop a method permitting such a partial regeneration. This **fast regeneration** process is microprocessor controlled and permits a partial regeneration of the cryopump in about 40 minutes compared to 6 hours needed for a total regeneration based on the purge gas method. A comparison between the typical cycles for total and partial regeneration is shown in Fig. 2.70. The time saved by the Fast Regeneration System is apparent. In a production environment for typical sputtering processes one will have to expect one

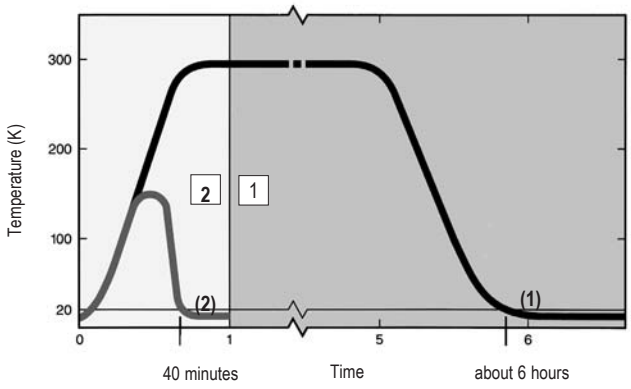


Fig. 2.70 Comparison between total (1) and partial (2) regeneration

total regeneration after 24 partial regenerations.

Throughput and **maximum pV flow:** (mbar l/s): the throughput of a cryopump for a certain gas depends on the pV flow of the gas G through the intake opening of the pump:

$Q_G = q_{pV,G}$; the following equation applies

$Q_G = p_G \cdot S_G$ with

p_G = intake pressure,
S_G = pumping capacity for the gas G

The maximum pV flow at which the cryopanels are warmed up to T ≈ 20 K in the case of **continuous operation**, depends on the net refrigerating power of the pump at this temperature and the type of gas. For refrigerator cryopumps and condensable gases the following may be taken as a guide:

$Q_{max} = 2.3\ \dot{Q}_2$ (20 K) mbar · l/s

$\dot{Q}_2$ (20 K) is the net refrigerating power in Watts available at the second stage of the cold heat at 20 K. In the case of **intermittent operation**, a higher pV flow is permissible (see crossover value).

Pumping speed S_{th}: The following applies to the (theoretical) pumping speed of a cryopump:

$$S_{th} = A_K \cdot S_A \cdot \alpha \cdot \left(1 - \frac{p_{end}}{p}\right) \qquad (2.29)$$

A_K Size of the cryopanels
S_A Surface area related pumping speed (area related impact rate according to equations 1.17 and 1.20, proportional to the mean velocity of the gas molecules in the direction of the cryopanel)
α Probability of condensation (pumping)
p_{end} Ultimate pressure (see above)
p Pressure in the vacuum chamber

The equation (2.29) applies to a cryopanel built into the vacuum chamber, the surface area of which is small compared to the surface of the vacuum chamber. At sufficiently low temperatures α = 1 for all gases. The equation (2.29) shows that for p >> pend the expression in brackets approaches 1 so that in the oversaturated case

p >> pend > Ps so that:

$$S_{th} = A_K \cdot S_A \qquad (2.29a)$$

with

$$S_A = \frac{\bar{c}}{4} = \sqrt{\frac{R \cdot T_G}{2 \cdot \pi \cdot M}} = 3.65\sqrt{\frac{T_G}{M}}\ \ell/s \cdot cm^2$$

T_G Gas temperature in K
M Molar mass

Given in Table 2.7 is the surface arearelated pumping speed S_A in $l \cdot s^{-1} \cdot cm^{-2}$ for some gases at two different gas temperatures T_G in K determined according to equation 2.29a. The values stated in the Table are limit values. In practice the condition of an almost undisturbed equilibrium

Symbol	Gas	M Molar mass	S_A at 293 K gas temp.	S_A at 80 K gas temp.	T_S Boiling point 1013 mbar	Triple point (= melting point)	
						T_t	p_t
		g/mol	l/s · cm²	l/s · cm²	K	K	mbar
H_2	Hydrogen	2.016	43.88	22.93	20.27	13.80	70.4
He	Helium	4.003	31.14	16.27	4.222	2.173	50.52
CH_4	Methane	4.003	15.56	8.13	111.67	90.67	116.7
H_2O	Water	18.015	14.68	–	373.15	273.16	6.09
Ne	Neon	20.183	13.87	7.25	27.102	24.559	433.0
CO	Carbon monoxide	28.000	11.77	6.15	81.67	68.09	153.7
N_2	Nitrogen	28.013	11.77	6.15	77.348	63.148	126.1
	Air	28.96	11.58	6.05	≈ 80.5	≈ 58.5	–
O_2	Oxygen	31.999	11.01	5.76	90.188	54.361	1.52
Ar	Argon	39.948	9.86	5.15	87.26	83.82	687.5
Kr	Krypton	83.80	6.81	3.56	119.4	115.94	713.9
Xe	Xenon	131.3	5.44	2.84	165.2	161.4	

Table 2.7 Surface-related pumping speeds for some gases

(small cryopanels compared to a large wall surface) is often not true, because large cryopanels are required to attain short pumpdown times and a good end vacuum. Deviations also result when the cryopanels are surrounded by a cooled baffle at which the velocity of the penetrating molecules is already reduced by cooling.

Service life / duration of operation t_{op} (s): The duration of operation of the cryopump for a particular gas depends on the equation:

$$C_G = \int_0^{t_{op,G}} Q_G(t)dt \quad \text{with}$$

C_G = Capacity of the cryopump for the gas G
$Q_G(t)$ = Throughput of the cryopump for the gas at the point of time t

If the constant mean over time for the throughput $\overline{Q_G}$ is known, the following applies:

$$t_{op,G} = \frac{C_G}{\overline{Q_G}} = \frac{C_G}{\overline{p_G} \cdot S_G} \qquad (2.30)$$

After the period of operation $t_{op,G}$ has elapsed the cryopump must be regenerated with respect to the type of gas G.

Starting pressure p_o: Basically it is possible to start a cryopump at atmospheric pressure. However, this is not desirable for several reasons. As long as the mean free path of the gas molecules is smaller than the dimensions of the vacuum chamber ($p > 10^{-3}$ mbar), thermal conductivity of the gas is so high that an unacceptably large amount of heat is transferred to the cryopanels. Further, a relatively thick layer of condensate would form on the cryopanel during starting. This would markedly reduce the capacity of the cryopump available to the actual operating phase. Gas (usually air) would be bonded to the adsorbent, since the bonding energy for this is lower than that for the condensation surfaces. This would further reduce the already limited capacity for hydrogen. It is recommended that cryopumps in the high vacuum or ultrahigh vacuum range are started with the aid of a backing pump at pressures of $p < 5 \cdot 10^{-2}$ mbar. As soon as the starting pressure has been attained the backing pump may be switched off.

2.2 Choice of pumping process

2.2.1 Survey of the most usual pumping processes

Vacuum technology has undergone rapid development since the Fifties. In research and in most branches of industry today, it is indispensable.

Corresponding to the many areas of application, the number of technical procedures in vacuum processes is extraordinarily large. These cannot be described within the scope of this section, because the basic calculations in this section cover mainly the pumping process, not the process taking place in the vessel. A survey of the most important processes in vacuum technology and the pressure regions in which these processes are chiefly carried out is given in the diagrams (Figures 2.71 and 2.72).

Generally, the pumping operation for these processes can be divided into two categories – dry- and wet – vacuum procedures, that is, into processes in which no significant amounts of vapor have to be pumped and those in which vapors (mostly water or organic) arise.

Distinctions between the two categories are described briefly:

Dry processes work primarily in a narrow and limited pressure region. The system is usually evacuated to a suitable characteristic pressure before the actual working process begins. This happens, for example, in plants for evaporative coating, electron-beam welding, and crystal pulling; in particle accelerators, mass spectrometers, electron microscopes; and others.

Further, there are dry processes in which degassing in vacuum is the actual technical process. These include work in induction- and arc furnaces, steel degassing plants, and plants for the manufacture of pure metals and electron tubes.

Wet processes are undertaken primarily in a prescribed working operation that covers a wider pressure region. This is especially important in the drying of solid materials. If, for instance, work is undertaken prematurely at too low a pressure, the outer surfaces dry out too quickly. As a result, the thermal contact to the moisture to be evaporated is impaired and the drying time is considerably increased. Predominantly processes that are carried out in drying, impregnating, and freeze-drying plants belong in this category.

In the removal of water vapor from liquids or in their distillation, particularly in degassing columns, vacuum filling, and resin-casting plants, as well as in molecular distillation, the production of as large a liquid surface as possible is important. **In all wet processes the provision of the necessary heat for evaporation of the moisture is of great importance.**

Basic pumping procedures are given in the following paragraphs.

If you have specific questions, you should get in touch with a specialist department in LEYBOLD where experts are available to you who can draw on many years of experience.

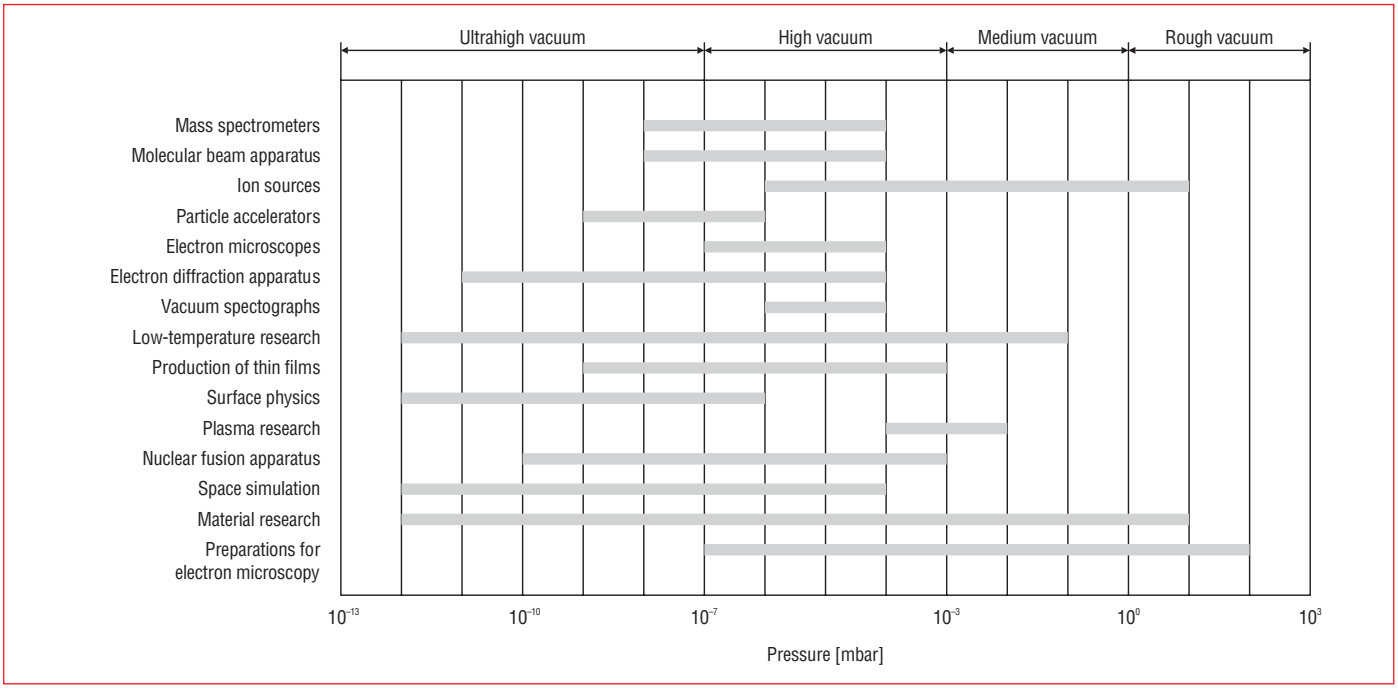


Fig. 2.71 Pressure ranges (p < 1000 mbar) of physical and chemical analytical methods

Classifications of typical vacuum processes and plants according to the pressure regions

Rough vacuum 1013 mbar – 1 mbar

- Drying, distillation, and steel degassing.

Medium vacuum 1 -10^{-3} mbar

- Molecular distillation, freeze-drying, impregnation, melting and casting furnaces, and arc furnaces.

High vacuum 10^{-3} – 10^{-7} mbar

- Evaporative coating, crystal pulling, mass spectrometers, tube production, electron microscopes, electron beam plants, and particle accelerators.

Ultrahigh vacuum: < 10^{-7} mbar

- Nuclear fusion, storage rings for accelerators, space research, and surface physics.

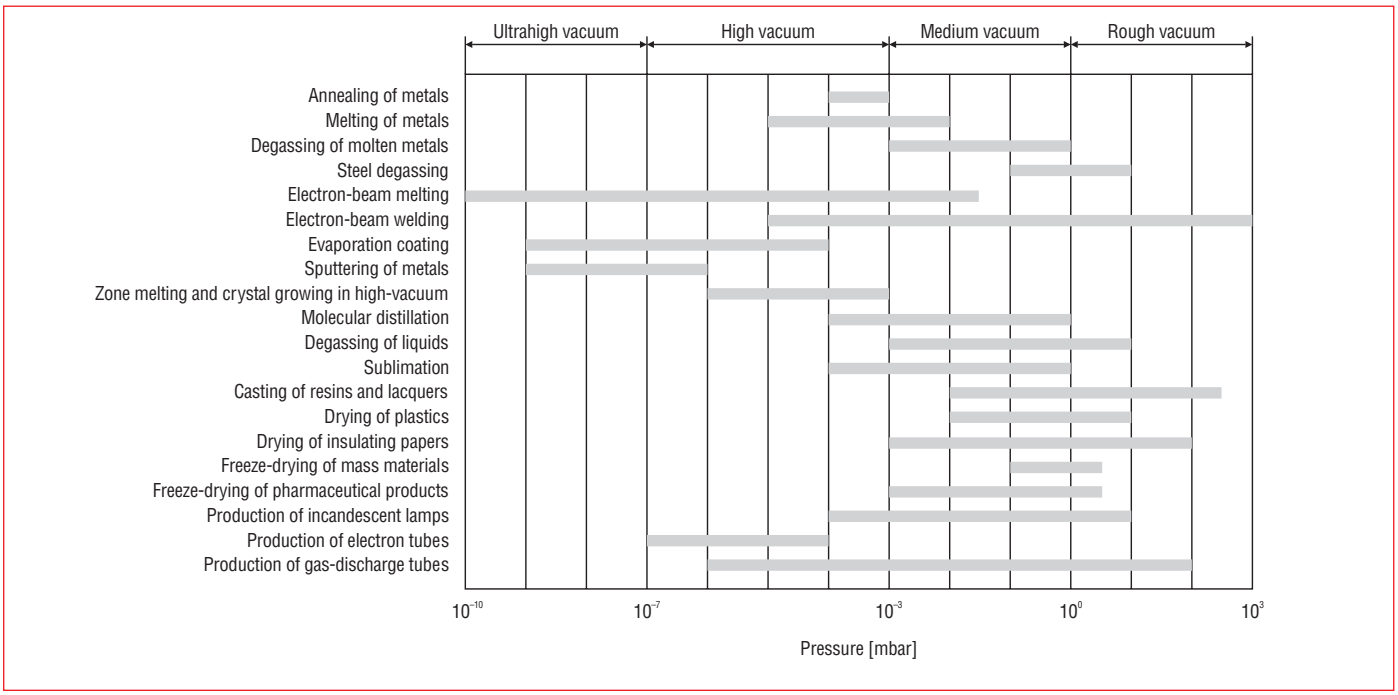


Fig. 2.72 Pressure ranges of industrial vacuum processes

2.2.2 Pumping of gases (dry processes)

For dry processes in which a non condensable gas mixture (e.g., air) is to be pumped, the pump to be used is clearly characterized by the required working pressure and the quantity of gas to be pumped away. The choice of the required working pressure is considered in this section. The choice of the required pump is dealt with in Section 2.3.

Each of the various pumps has a characteristic working range in which it has a particularly high efficiency. Therefore, the most suitable pumps for use in the following individual pressure regions are described. For every dry-vacuum process, the vessel must first be evacuated. It is quite possible that the pumps used for this may be different from those that are the optimum choices for a process that is undertaken at definite working pressures. In every case the choice should be made with particular consideration for the pressure region in which the working process predominantly occurs.

a) Rough vacuum (1013 – 1 mbar)
The usual working region of the rotary pumps described in Section 2 lies below 80 mbar. At higher pressures these pumps have a very high power consumption (see Fig. 2.11) and a high oil consumption (see Section 8.3.1.1). Therefore, if gases are to be pumped above 80 mbar over long periods, one should use, particularly on economic grounds, jet pumps, water ring pumps or dry running, multi-vane pumps. Rotary vane and rotary piston pumps are especially suitable for pumping down vessels from atmospheric pressure to pressures below 80 mbar, so that they can work continuously at low pressures. If large quantities of gas arise at inlet pressures below 40 mbar, the connection in series of a Roots pump is recommended. Then, for the backing pump speed required for the process concerned, a much smaller rotary vane or piston pump can be used.

b) Medium vacuum (1 – 10^{-3} mbar)
If a vacuum vessel is merely to be evacuated to pressures in the medium vacuum region, perhaps to that of the required backing pressure for diffusion or sputter-ion pumps, single- and two-stage rotary pumps are adequate for pressures down to 10^{-1} and 10^{-3} mbar, respectively. It is essentially more difficult to select the suitable type of pump if medium vacuum processes are concerned in which gases or vapors are evolved continuously and must be pumped away. An important hint may be given at this point. Close to the attainable ultimate pressure, the pumping speed of all rotary pumps falls off rapidly. Therefore, the lowest limit for the normal working pressure region of these pumps should be that at which the pumping speed still amounts to about 50 % of the nominal pumping speed.

Between 1 and 10^{-2} mbar at the onset of large quantities of gas, Roots pumps with rotary pumps as backing pumps have optimum pumping properties (see Section 2.1.3.1). For this pressure range, a single-stage rotary pump is sufficient, if the chief working region lies above 10^{-1} mbar. If it lies between 10^{-1} and 10^{-2} mbar, a two-stage backing pump is recommended. Below 10^{-2} mbar the pumping speed of single-stage Roots pumps in combination with two-stage rotary pumps as backing pumps decreases. However, between 10^{-2} and 10^{-4} mbar, two-stage Roots pumps (or two single-stage Roots pumps in series) with two-stage rotary pumps as backing pumps still have a very high pumping speed. Conversely, this pressure region is the usual working region for vapor ejector pumps. For work in this pressure region, they are the most economical pumps to purchase. As backing pumps, single-stage rotary positive displacement pumps are suitable. If very little maintenance and valveless operation are convenient (i.e., small vessels in short operation cycles are to be pumped to about 10^{-4} mbar or large vessels are to be maintained at this pressure unattended for weeks), the previously mentioned two-stage Roots pumps with two-stage rotary pumps as backing pumps are the suitable combinations. Allthough, such a combination does not work as economically as the corresponding vapor ejector pump, it can operate for a much longer time without maintenance.

c) High vacuum (10^{-3} to 10^{-7} mbar)
Diffusion, sputter-ion, and turbomolecular pumps typically operate in the pressure region below 10^{-3} mbar. If the working region varies during a process, different pumping systems must be fitted to the vessel. There are also special diffusion pumps that combine the typical properties of a diffusion pump (low ultimate pressure, high pumping speed in the high vacuum region) with the outstanding properties of a vapor ejector pump (high throughput in the medium vacuum region, high critical backing pressure). If the working region lies between 10^{-2} and 10^{-6} mbar, these diffusion pumps are, in general, specially recommended.

d) Ultrahigh vacuum (< 10^{-7} mbar)
For the production of pressures in the ultrahigh vacuum region, sputter-ion, and sublimation pumps, as well as turbomolecular pumps and cryopumps, are used in combination with suitable forepumps. The pump best suited to a particular UHV process depends on various conditions (for further details, see Section 2.5).

2.2.3 Pumping of gases and vapors (wet processes)

When vapors must be pumped, in addition to the factors working pressure and pumping speed, a third determining factor is added namely the vapor partial pressure – which may vary considerably in the course of a process. This factor is decisive in determining the pumping arrangement to be installed. In this regard, the condensers described in Section 2.15 are very important adjuncts to rotary displacement pumps. They have a particularly high pumping speed when pumping vapors. The next section covers pumping of water vapor (the most frequent case). The considerations apply similarly to other non-aggressive vapors.

Pumping of Water Vapor
Water vapor is frequently removed by pumps that operate with water or steam as a pump fluid, for example, water ring pumps or steam ejector pumps. This depends considerably on circumstances, however, because the economy of steam ejector pumps at low pressures is generally far inferior to that of rotary pumps. For pumping a vapor – gas mixture in which the vapor portion is large but the air portion is small, the vapor can be pumped by condensers and the permanent gases, by relatively small gas ballast pumps (see Section 2.1.5).

Comparatively, then, a pump set consisting of a Roots pump, condenser, and backing pump, which can transport 100 kg/h of vapor and 18 kg/h of air at an inlet pressure of 50 mbar, has a power requirement of 4 – 10 kW (depending on the quantity of air involved). A steam ejector pump of the same performance requires about 60 kW without altering the quantity of air involved.

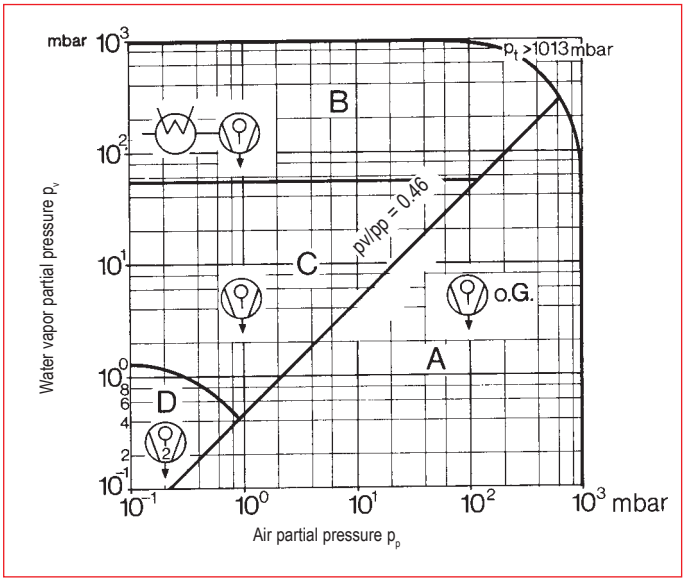


Fig. 2.73 Areas of application for gas ballast pumps and condensers pumping water vapor (o.G. = without gas ballast)

For the pumping of water vapor, gas ballast pumps and combinations of gas ballast pumps, Roots pumps, and condensers are especially suitable.

Pumping of water vapor with gas ballast pumps

The ratio of vapor partial pressure p_v to air partial pressure p_p is decisive in the evaluation of the correct arrangement of gas ballast pumps, as shown previously by equations 2.2 and 2.3. Therefore, if the water vapor tolerance of the gas ballast pump is known, graphs may be obtained that clearly give the correct use of gas ballast pumps for pumping water vapor (see Fig. 2.73). Large single-stage rotary plunger pumps have, in general, an operating temperature of about 77 °C and hence a water vapor tolerance of about 60 mbar. This value is used to determine the different operating regions in Fig. 2.73. In addition it is assumed that the pressure at the discharge outlet port of the gas ballast pump can increase to a maximum of 1330 mbar until the discharge outlet valve opens.

Region A: Single-stage, rotary plunger pumps without gas ballast inlet.

At a saturation vapor pressure p_S of 419 mbar at 77 °C, according to equation 2.2, the requirement is given that $p_v < 0.46\ p_p$, where

p_v is the water vapor partial pressure
p_p is the partial pressure of air
$p_v + p_p = p_{tot}$ total pressure

This requirement is valid in the whole working region of the single-stage rotary plunger pump – hence, at total pressures between 10^{-1} and 1013 mbar.

Region B: Single-stage rotary plunger pumps with gas ballast and an inlet condenser.

In this region the water vapor pressure exceeds the admissible partial pressure at the inlet. The gas ballast pump must, therefore, have a condenser inserted at the inlet, which is so rated that the water vapor partial pressure at the inlet port of the rotary pump does not exceed the admissible value. The correct dimensions of the condenser are selected depending on the quantity of water vapor involved. For further details, see Section 2.1.5.

At a water vapor tolerance of 60 mbar, the lower limit of this region is

$$p_v > 6O + 0.46\ p_p \text{ mbar}$$

Region C: Single-stage rotary plunger pumps with a gas ballast.

The lower limit of region C is characterized by the lower limit of the working region of this pump. It lies, therefore, at about ptot = 1 mbar. If large quantities of vapor arise in this region, it is often more economical to insert a condenser: 20 kg of vapor at 28 mbar results in a volume of about 1000 m^3. It is not sensible to pump this volume with a rotary pump. As a rule of thumb:

A condenser should always be inserted at the pump's inlet if saturated water vapor arises for a considerable time.

As a precaution, therefore, a Roots pump should always be inserted in front of the condenser at low inlet pressures so that the condensation capacity is essentially enhanced. The condensation capacity does not depend only on the vapor pressure, but also on the refrigerant temperature. At low vapor pressures, therefore, effective condensation can be obtained only if the refrigerant temperature is correspondingly low. At vapor pressures below 6.5 mbar, for example, the insertion of a condenser is sensible only if the refrigerant temperature is less than 0 °C. Often at low pressures a gas – vapor mixture with unsaturated water vapor is pumped (for further details, see Section 2.1.5). In general, then, one can dispense with the condenser.

Region D: Two-stage gas ballast pumps, Roots pumps, and vapor ejector pumps, always according to the total pressure concerned in the process.

It must again be noted that the water vapor tolerance of two-stage gas ballast pumps is frequently lower than that of corresponding single-stage pumps.

Pumping of water vapor with roots pumps

Normally, Roots pumps are not as economical as gas ballast pumps for continuous operation at pressures above 40 mbar. With very large pump sets, which work with very specialized gear ratios and are provided with bypass lines, however, the specific energy consumption is indeed more favorable. If Roots pumps are installed to pump vapors, as in the case of gas ballast pumps, a chart can be given that includes all possible cases (see Fig. 2.74).

Region A: A Roots pump with a single-stage rotary plunger pump without gas ballast.

As there is merely a compression between the Roots pump and the rotary plunger pump, the following applies here too:

$$p_v < 0.46\ p_p$$

The requirement is valid over the entire working region of the pump combination and, therefore, for total pressures between 10^{-2} and 40 mbar (or 1013 mbar for Roots pumps with a bypass line).

Region B: A main condenser, a Roots pump with a bypass line, an intermediate condenser, and a gas ballast pump.

This combination is economical only if large water vapor quantities are to be pumped continuously at inlet pressures above about 40 mbar. The size of the main condenser depends on the quantity of vapor involved.

Vacuum generation

The intermediate condenser must decrease the vapor partial pressure below 60 mbar. Hence, the gas ballast pump should be large enough only to prevent the air partial pressure behind the intermediate condenser from exceeding a certain value; for example, if the total pressure behind the Roots pump (which is always equal to the total pressure behind the intermediate condenser) is 133 mbar, the gas ballast pump must pump at least at a partial air pressure of 73 mbar, the quantity of air transported to it by the Roots pump. Otherwise, it must take in more water vapor than it can tolerate. This is a basic requirement: **the use of gas ballast pumps is wise only if air is also pumped**!

With an ideally leak-free vessel, the gas ballast pump should be isolated after the required operating pressure is reached and pumping continued with the condenser only. Section 2.1.5 explains the best possible combination of pumps and condensers.

Region C: A Roots pump, an intermediate condenser, and a gas ballast pump.
The lower limit of the water vapor partial pressure is determined through the compression ratio of the Roots pump at the backing pressure, which is determined by the saturation vapor pressure of the condensed water. Also, in this region the intermediate condenser must be able to reduce the vapor partial pressure to at least 60 mbar. The stated arrangement is suitable – when cooling the condenser with water at 15 °C – for water vapor pressures between about 4 and 40 mbar.

Region D: A roots pump and a gas ballast pump.
In this region D the limits also depend essentially on the stages and ratios of sizes of the pumps. In general, however, this combination can always be used between the previously discussed limits – therefore, between 10^{-2} and 4 mbar.

2.2.4 Drying processes

Often a vacuum process covers several of the regions quoted here. In batch drying the process can, for example (see Fig. 2.74), begin in region A (evacuation of the empty vessel) and then move through regions B, C, and D in steps. Then the course of the process would be as follows:

A. Evacuating the vessel by a gas ballast pump and a Roots pump with a bypass line.

B. Connecting the two condensers because of the increasing vapor pressure produced by heating the material.
The choice of the pumping System is decided by the highest vapor partial pressure occurring and the lowest air partial pressure at the inlet.

C. Bypassing the main condenser
It will now not have an effect. Instead it would only be pumped empty by the pumping system with a further drop in vapor pressure.

D. Bypassing the intermediate condenser.
Roots pumps and gas ballast pumps alone can now continue pumping. With short-term drying, the separation of the condenser filled with condensed water is particularly important, because the gas ballast pump would continue to pump from the condenser the previously condensed water vapor at the saturation vapor pressure of water.

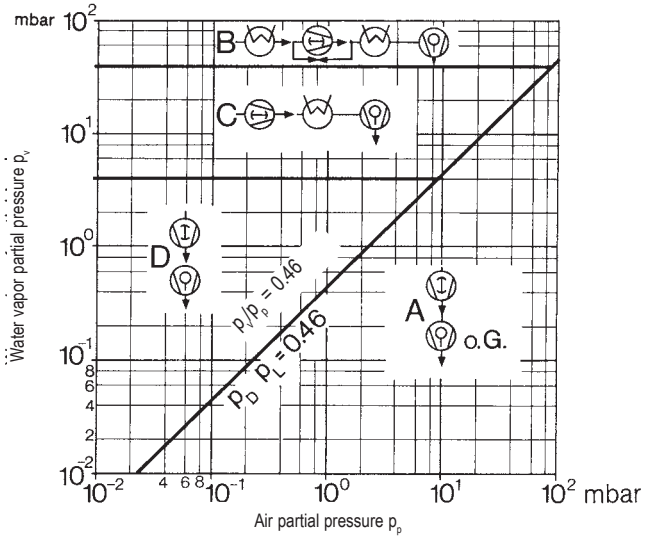


Fig. 2.74 Areas of application for Roots pumps and condensers pumping water vapor (o.G. = without gas ballast)

With longer-term drying processes, it suffices to shut off the condensate collector from the condenser. Then only the remaining condensate film on the cooling tubes can reevaporate. Depending on the size of the gas ballast pump, this reevaporation ensues in 30 – 60 min.

E. If the drying process should terminate at still lower pressures
When a pressure below 10^{-2} mbar is reached a previously bypassed oil vapor ejector pump should be switched on in addition.

Drying of solid substances
As previously indicated, the drying of solid substances brings about a series of further problems. It no longer suffices that one simply pumps out a vessel and then waits until the water vapor diffuses from the solid substance. This method is indeed technically possible, but it would intolerably increase the drying time.

It is not a simple technical procedure to keep the drying time as short as possible. Both the water content and the layer thickness of the drying substance are important. Only the principles can be stated here. In case of special questions we advise you to contact our experts in our Cologne factory.

The moisture content E of a material to be dried of which the diffusion coefficient depends on the moisture content (e.g. with plastics) as a function of the drying time t is given in close approximation by the following equation:

$$E = \frac{E_0}{(1 + K \cdot t)^q} \% \qquad (2.31)$$

E_0 where E is the moisture content before drying
q is the temperature-dependent coefficient. Thus equation (2.31) serves only for the temperature at which q was determined
K is a factor that depends on the temperature, the water vapor partial pressure in the vicinity of the material, the dimensions, and the properties of the material.

With the aid of this approximate equation, the drying characteristics of many substances can be assessed. If K and q have been determined for various temperatures and water vapor partial pressures, the values for other temperatures are easily interpolated, so that the course of the drying process can be calculated under all operating conditions. With the aid of a similarity transformation, one can further compare the course of drying process of a material with known properties with that of a material with different properties.

Fundamentally, in the drying of a material, a few rules are noteworthy:
Experience has shown that shorter drying times are obtained if the water vapor partial pressure at the surface of the material is relatively high, that is, if the surface of the material to be dried is not yet fully free of moisture. This is possible because the heat conduction between the source of heat and the material is greater at higher pressures and the resistance to diffusion in a moist surface layer is smaller than in a dry one. To fulfill the conditions of a moist surface, the pressure in the drying chamber is controlled. If the necessary relatively high water vapor partial pressure cannot be maintained permanently, the operation of the condenser is temporarily discontinued. The pressure in the chamber then increases and the surface of the material becomes moist again. To reduce the water vapor partial pressure in the vessel in a controlled way, it may be possible to regulate the refrigerant temperature in the condenser. In this way, the condenser temperature attains preset values, and the water vapor partial pressure can be reduced in a controlled manner.

2.2.5 Production of an oil-free (hydrocarbon-free) vacuum

Backstreaming vapor pump fluids, vapors of oils, rotary pump lubricants, and their cracking products can significantly disturb various working processes in vacuum. Therefore, it is recommended that certain applications use pumps and devices that reliably exclude the presence of hydrocarbon vapors.

a) Rough vacuum region (1013 to 1 mbar)
Instead of rotary pumps, large water jet, steam ejector, or water ring pumps can be used. For batch evacuation, and the production of hydrocarbon-free fore vacuum for sputter-ion pumps, adsorption pumps (see Section 2.1.8.1) are suitable. If the use of oil-sealed rotary vane pumps cannot be avoided, basically two-stage rotary vane pumps should be used. The small amount of oil vapor that backstreams out of the inlet ports of these pumps can be almost completely removed by a sorption trap (see Section 2.1.4) inserted in the pumping line.

b) Medium vacuum region (1 to 10^{-3} mbar)
For the pumping of large quantities of gas in this pressure region, vapor ejector pumps are by far the most suitable. With mercury vapor ejector pumps, completely oil-free vacua can be produced. As a precaution, the insertion of a cold trap chilled with liquid nitrogen is recommended so that the harmful mercury vapor does not enter the vessel. With the medium vacuum sorption traps described under a), it is possible with two-stage rotary vane pumps to produce almost oil-free vacua down to below 10^{-4} mbar.

Absolutely oil-free vacua may be produced in the medium vacuum region with adsorption pumps. Since the pumping action of these pumps for the light noble gases is only small, vessels initially filled with air can only be evacuated by them to about 10^{-2} mbar. Pressures of 10^{-3} mbar or lower can then be produced with adsorption pumps only if neither neon nor helium is present in the gas mixture to be pumped. In such cases it can be useful to expel the air in the vessel by first flooding with nitrogen and then pumping it away.

c) High- and ultrahigh vacuum region (< 10^{-3} mbar)
When there is significant evolution of gas in the pressure regions that must be pumped, turbomolecular pumps, or cryopumps should be used. A sputter-ion pump is especially suitable for maintaining the lowest possible pressure for long periods in a sealed system where the process does not release large quantities of gas. Magnetically-suspended turbomolecular pumps also guarantee hydrocarbon-free vacua. However, while these pumps are switched off, oil vapors can enter the vessel through the pump. By suitable means (e.g., using an isolating valve or venting the vessel with argon), contamination of the vessel walls can be impeded when the pump is stationary. If the emphasis is on generating a “hydrocarbon-free vacuum” with turbomolecular pumps, then hybrid turbomolecular pumps with diaphragm pumps or classic turbomolecular pumps combined with scroll pumps should be used as oil-free backing pumps.

2.2.6 Ultrahigh vacuum working Techniques

The boundary between the high and ultrahigh vacuum region cannot be precisely defined with regard to the working methods. In practice, a border between the two regions is brought about because pressures in the high vacuum region may be obtained by the usual pumps, valves, seals, and other components, whereas for pressures in the UHV region, another technology and differently constructed components are generally required. The “border” lies at a few 10^{-8} mbar. Therefore, pressures below 10^{-7} mbar should generally be associated with the UHV region.

The gas density is very small in the UHV region and is significantly influenced by outgasing rate of the vessel walls and by the tiniest leakages at joints. Moreover, in connection with a series of important technical applications to characterize the UHV region, generally the monolayer time (see also equation 1.21) has become important. This is understood as the time τ that elapses before a monomolecular or monatomic layer forms on an initially ideally cleaned surface that is exposed to the gas particles. Assuming that every gas particle that arrives at the surface finds a free place and remains there, a convenient formula for τ is

$$\tau = \frac{3.2}{p} \cdot 10^{-6}\ \mathrm{s} \qquad \text{(p in mbar)}$$

Therefore, in UHV ($p < 10^{-7}$ mbar) the monolayer formation time is of the order of minutes to hours or longer and thus of the same length of time as that needed for experiments and processes in vacuum. The practical requirements that arise have become particularly significant in solid-state physics, such as for the study of thin films or electron tube technology. A UHV system is different from the usual high vacuum system for the following reasons:

a) the leak rate is extremely small (use of metallic seals),

b) the gas evolution of the inner surfaces of the vacuum vessel and of the attached components (e.g., connecting tubulation; valves, seals) can be made extremely small,

c) suitable means (cold traps, baffles) are provided to prevent gases or vapors or their reaction products that have originated from the pumps used from reaching the vacuum vessel (no backstreaming).

To fulfill these conditions, the individual components used in UHV apparatus must be bakeable and extremely leaktight. Stainless steel is the preferred material for UHV components.

The construction, start-up, and operation of an UHV system also demands special care, cleanliness, and, above all, time. The assembly must be appropriate; that is, the individual components must not be in the least damaged (i.e. by scratches on precision-worked sealing surfaces). Fundamentally, every newly-assembled UHV apparatus must be tested for leaks with a helium leak detector before it is operated. Especially important here is the testing of demountable joints (flange connections), glass seals, and welded or brazed joints. **After testing, the UHV apparatus must be baked out.** This is necessary for glass as well as for metal apparatus. The bake-out extends not only over the vacuum vessel, but frequently also to the attached parts, particularly the vacuum gauges. The individual stages of the bake-out, which can last many hours for a larger system, and the bake-out temperature are arranged according to the kind of plant and the ultimate pressure required. If, after the apparatus has been cooled and the other necessary measures undertaken (e.g., cooling down cold traps or baffles), the ultimate pressure is apparently not obtained, a repeated leak test with a helium leak detector is recommended. Details on the components, sealing methods and vacuum gauges are provided in our catalog.

2.3. Evacuation of a vacuum chamber and determination of pump sizes

Basically, two independent questions arise concerning the size of a vacuum system:

1. What effective pumping speed must the pump arrangement maintain to reduce the pressure in a given vessel over a given time to a desired value?

2. What effective pumping speed must the pump arrangement reach during a vacuum process so that gases and vapors released into the vessel can be quickly pumped away while a given pressure (the operating pressure) in the vessel, is maintained and not exceeded?

During the pumping-out procedure of certain processes (e.g., drying and heating), vapors are produced that were not originally present in the vacuum chamber, so that a third question arises:

3. What effective pumping speed must the pump arrangement reach so that the process can be completed within a certain time?

The **effective pumping speed** of a pump arrangement is understood as the **actual pumping speed** of the entire pump arrangement **that prevails at the vessel**. The nominal pumping speed of the pump can then be determined from the effective pumping speed if the flow resistance (conductances) of the baffles, cold traps, filters, valves, and tubulations installed between the pump and the vessel are known (see Sections 1.5.2 to 1.5.4). In the determination of the required nominal pumping speed it is further assumed that the vacuum system is leaktight; therefore, the leak rate must be so small that gases flowing in from outside are immediately removed by the connected pump arrangement and the pressure in the vessel does not alter (for further details, see Section 5). The questions listed above under 1., 2. and 3. are characteristic for the three most essential exercises of vacuum technology

1. Evacuation of the vessel to reach a specified pressure.

2. Pumping of continuously evolving quantities of gas and vapor at a certain pressure.

3. Pumping of the gases and vapors produced during a process by variation of temperature and pressure.

Initial evacuation of a vacuum chamber is influenced in the medium-, high-, and ultrahigh vacuum regions by continually evolving quantities of gas, because in these regions the escape of gases and vapors from the walls of the vessel is so significant that they alone determine the dimensions and layout of the vacuum system.

2.3.1 Evacuation of a vacuum chamber (without additional sources of gas or vapor)

Because of the factors described above, an assessment of the pump-down time must be basically different for the evacuation of a container in the rough vacuum region from evacuation in the medium- and high vacuum regions.

2.3.1.1 Evacuation of a chamber in the rough vacuum region

In this case the required effective pumping speed S_{eff}, of a vacuum pump assembly is dependent only on the required pressure p, the volume V of the container, and the pump-down time t.

With constant pumping speed S_{eff} and assuming that the ultimate pressure p_{end} attainable with the pump arrangement is such that $p_{end} << p$, the decrease with time of the pressure p(t) in a chamber is given by the equation:

$$-\frac{dp}{dt} = \frac{S_{eff}}{V} \cdot p \qquad (2.32)$$

Beginning at 1013 mbar at time t = 0, the effective pumping speed is calculated depending on the pump-down time t from equation (2.32) as follows:

$$\int_{1013}^{p} \frac{dp}{p} = -\frac{S_{eff}}{V} \cdot t \qquad (2.33a)$$

$$\ell n \frac{p}{1013} = -\frac{S_{eff}}{V} \cdot t \qquad (2.33b)$$

$$S_{eff} = \frac{V}{t} \cdot \ell n \frac{1013}{p} = \frac{V}{t} \cdot 2.3 \cdot \log \frac{1013}{p} \qquad (2.34)$$

Introducing the dimensionless factor

$$\sigma = \ell n \frac{1013}{p} = 2.3 \cdot \log \frac{1013}{p} \qquad (2.34a)$$

into equation (2.34), the relationship between the effective pumping speed S_{eff}, and the pump-down time t is given by

$$S_{eff} = \frac{V}{t} \cdot \sigma \qquad (2.35)$$

The ratio V/S_{eff} is generally designated as a time constant τ. Thus the pump-down time of a vacuum chamber from atmospheric pressure to a pressure p is given by:

$$t = \tau \cdot \sigma \qquad (2.36)$$

with $\tau = \frac{V}{S_{eff}}$

and $\sigma = \ell n \frac{1013}{p}$

The dependence of the factor from the desired pressure is shown in Fig. 2.75. It should be noted that the pumping speed of single-stage rotary vane and rotary piston pumps decreases below 10 mbar with gas ballast and below 1 mbar without gas ballast. This fundamental behavior is different for pumps of various sizes and types but should not be ignored in the determination of the dependence of the pump-down time on pump size. It must be pointed out that the equations (2.32 to 2.36) as well Fig. 2.75 only apply when the ultimate pressure attained with the pump used is by several orders of magnitude lower than the desired pressure.

Example: A vacuum chamber having a volume of 500 l shall be pumped down to 1 mbar within 10 minutes. What effective pumping speed is required?

500 l = 0.5 m^3; 10 min = 1/6 h

According to equation (2.34) it follows that:

$$S_{eff} = \frac{0.5}{1/6} \cdot 2.3 \cdot \log \frac{1013}{1}$$
$$= 3 \cdot 2.3 \cdot 3.01 = 20.8\ m^3/h$$

For the example given above one reads off the value of 7 from the straight line in Fig. 2.75. However, from the broken line a value of 8 is read off. According to equation (2.35) the following is obtained:

$$S_{eff} = \frac{0.5}{\frac{1}{6}} \cdot 7 = 21\ m^3/h \quad \text{or}$$

$$S_{eff} = \frac{0.5}{\frac{1}{6}} \cdot 8 = 24\ m^3/h$$

under consideration of the fact that the pumping speed reduces below 10 mbar. The required effective pumping speed thus amounts to about 24 m^3/h.

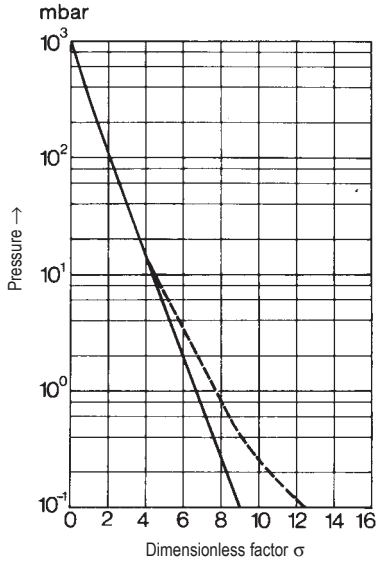


Fig. 2.75 Dependency of the dimensionless factor s for calculation of pumpdown time t according to equation 2.36. The broken line applies to single-stage pumps where the pumping speed decreases below 10 mbar.

2.3.1.2 Evacuation of a chamber in the high vacuum region

It is considerably more difficult to give general formulas for use in the high vacuum region. Since the pumping time to reach a given high vacuum pressure depends essentially on the gas evolution from the chamber's inner surfaces, the condition and pre-treatment of these surfaces are of great significance in vacuum technology. Under no circumstances should the material used exhibit porous regions or – particularly with regard to bake-out – contain cavities; the inner surfaces must be as smooth as possible (true surface = geometric surface) and thoroughly cleaned (and degreased). Gas evolution varies greatly with the choice of material and the surface condition. Useful data are collected in Table X (Section 9). The gas evolution can be determined experimentally only from case to case by the **pressure-rise method**: the system is evacuated as thoroughly as possible, and finally the pump and the chamber are isolated by a valve. Now the time is measured for the pressure within the chamber (volume V) to rise by a certain amount, for example, a power of 10. The gas quantity Q that arises per unit time is calculated from:

$$Q = \frac{\Delta p \cdot V}{t} \qquad (2.37)$$

(Δp = measured pressure rise)

The gas quantity Q consists of the sum of all the gas evolution and all leaks possibly present. Whether it is from gas evolution or leakage may be determined by the following method:

The gas quantity arising from gas evolution must become smaller with time, the quantity of gas entering the system from leakage remains constant with time. Experimentally, this distinction is not always easily made, since it often takes a considerable length of time – with pure gas evolution – before the measured pressure-time curve approaches a constant (or almost a constant) final value; thus the beginning of this curve follows a straight line for long times and so simulates leakage (see Section 5, Leaks and Leak Detection).

If the gas evolution Q and the required pressure p_{end} are known, it is easy to determine the necessary effective pumping speed:

$$S_{eff} = \frac{Q}{p_{end}} \qquad (2.38)$$

Example: A vacuum chamber of 500 l may have a total surface area (including all systems) of about 5 m^2. A steady gas evolution of $2 \cdot 10^{-4}$ mbar · l/s is assumed per m^2 of surface area. This is a level which is to be expected when valves or rotary feedthroughs, for example are connected to the vacuum chamber. In order to maintain in the system a pressure of $1 \cdot 10^{-5}$ mbar, the pump must have a pumping speed of

$$S_{eff} = \frac{5 \cdot 2 \cdot 10^{-4}\ \text{mbar} \cdot \ell/\text{s}}{1 \cdot 10^{-5}\ \text{mbar}} = 100\,\ell/\text{s}$$

A pumping speed of 100 l/s alone is required to continuously pump away the quantity of gas flowing in through the leaks or evolving from the chamber walls. Here the evacuation process is similar to the examples given in Sections 2.3.1.1. However, in the case of a diffusion pump the pumping process does not begin at atmospheric pressure but at the forevacuum pressure pV instead. Then equation (2.34) transforms into:

$$S_{eff} = \frac{V}{t} \cdot \ell n \frac{p_V}{p} = \frac{V}{t} \cdot \ell n\, K$$

At a backing pressure of $p_V = 2 \cdot 10^{-3}$ mbar "compression" K is in our example:

$$K = \frac{2 \cdot 10^{-3}}{1 \cdot 10^{-5}} = 200$$

In order to attain an ultimate pressure of $1 \cdot 10^{-5}$ mbar within 5 minutes after starting to pump with the diffusion pump an effective pumping speed of

$$S_{eff} = \frac{500}{5 \cdot 60} \cdot 2.3 \cdot \log 200 \approx 9\,\frac{\ell}{\text{s}}$$

is required. This is much less compared to the effective pumping speed needed to maintain the ultimate pressure. Pumpdown time and ultimate vacuum in the high vacuum and ultrahigh vacuum ranges depends mostly on the gas evolution rate and the leak rates. The underlying mathematical rules can not be covered here. For these please refer to books specializing on that topic.

2.3.1.3 Evacuation of a chamber in the medium vacuum region

In the rough vacuum region, the volume of the vessel is decisive for the time involved in the pumping process. In the high and ultrahigh vacuum regions, however, the gas evolution from the walls plays a significant role. In the medium vacuum region, the pumping process is influenced by both quantities. Moreover, in the medium vacuum region, particularly with rotary pumps, the ultimate pressure pend attainable is no longer negligible. If the quantity of gas entering the chamber is known to be at a rate Q (in millibars liter per second) from gas evolution from the walls and leakage, the differential equation (2.32) for the pumping process becomes

$$\frac{dp}{dt} = -\frac{S_{eff}\left(p - p_{end}\right) - Q}{V} \qquad (2.39)$$

Integration of this equation leads to

$$t = \frac{V}{S_{eff}}\, \ell n \frac{\left(p_o - p_{end}\right) - Q/S_{eff}}{\left(p - p_{end}\right) - Q/S_{eff}} \qquad (2.40)$$

where

p_0 is the pressure at the beginning of the pumping process
p is the desired pressure

In contrast to equation 2.33b this equation does not permit a definite solution for S_{eff}, therefore, the effective pumping speed for a known gas evolution cannot be determined from the time – pressure curve without further information.

In practice, therefore, the following method will determine a pump with sufficiently high pumping speed:

a) The pumping speed is calculated from equation 2.34 as a result of the volume of the chamber without gas evolution and the desired pump-down time.

b) The quotient of the gas evolution rate and this pumping speed is found. This quotient must be smaller than the required pressure; for safety, it must be about ten times lower. If this condition is not fulfilled, a pump with correspondingly higher pumping speed must be chosen.

2.3.2 Determination of a suitable backing pump

The gas or vapor quantity transported through a high vacuum pump must also be handled by the backing pump. Moreover, in the operation of the high vacuum pump (diffusion pump, turbomolecular pump), the maximum permissible backing pressure must never, even for a short time, be exceeded. If Q is the effective quantity of gas or vapor, which is pumped by the high vacuum pump with an effective pumping speed S_{eff} at an inlet pressure p_A, this gas quantity must certainly be transported by the backing pump at a pumping speed of S_V at the backing pressure p_V. For the effective throughput Q, the continuity equation applies:

$$Q = p_A \cdot S_{eff} = pv \cdot S_V \qquad (2.41)$$

The required pumping speed of the backing pump is calculated from:

$$S_V = \frac{p_A}{p_V} \cdot S_{eff} \qquad (2.41a)$$

Example: In the case of a diffusion pump having a pumping speed of 400 l/s the effective pumping speed is 50 % of the value stated in the catalog when using a shell baffle. The max. permissible backing pressure is $2 \cdot 10^{-1}$ mbar. The pumping speed required as a minimum for the backing pump depends on the intake pressure p_A according to equation 2.41a.

At an intake pressure of $p_A = 1 \cdot 10^{-2}$ mbar the pumping speed for the high vacuum pump as stated in the catalog is about 100 l/s, subsequently 50 % of this is 50 l/s. Therefore the pumping speed of the backing pump must amount to at least

$$S_V = \frac{1 \cdot 10^{-2}}{2 \cdot 10^{-1}} \cdot 50 = 2.5\,\ell/s = 9\,m^3/h$$

At an intake pressure of $p_A = 1 \cdot 10^{-3}$ mbar the pump has already reached its nominal pumping speed of 400 l/s; the effective pumping speed is now S_{eff} = 200 l/s; thus the required pumping speed for the backing pump amounts to

$$S_V = \frac{1 \cdot 10^{-3}}{2 \cdot 10^{-1}} \cdot 200 = 1\,\ell/s = 3.6\,m^3/h$$

If the high vacuum pump is to be used for pumping of vapors between 10^{-3} and 10^{-2} mbar, then a backing pump offering a nominal pumping speed of 12 m^3/h must be used, which in any case must have a pumping speed of 9 m^3/h at a pressure of $2 \cdot 10^{-1}$ mbar. If no vapors are to be pumped, a single-stage rotary vane pump operated without gas ballast will do in most cases. If (even slight) components of vapor are also to be pumped, one should in any case use a two-stage gas ballast pump as the backing pump which offers – also with gas ballast – the required pumping speed at $2 \cdot 10^{-1}$ mbar.

If the high vacuum pump is only to be used at intake pressures below 10^{-3} mbar, a smaller backing pump will do; in the case of the example given this will be a pump offering a pumping speed of 6 m^3/h. If the continuous intake pressures are even lower, below 10^{-4} mbar, for example, the required pumping speed for the backing pump can be calculated from equation 2.41a as:

$$S_V = \frac{1 \cdot 10^{-4}}{2 \cdot 10^{-1}} \cdot 200 = 0.1\,\ell/s = 0.36\,m^3/h$$

Theoretically in this case a smaller backing pump having a pumping speed of about 1 m^3/h could be used. But in practice a larger backing pump should be installed because, especially when starting up a vacuum system, large amounts of gas may occur for brief periods. Operation of the high vacuum pump is endangered if the quantities of gas can not be pumped away immediately by the backing pump. If one works permanently at very low inlet pressures, the installation of a ballast volume (backing-line vessel or surge vessel) between the high vacuum pump and the backing pump is recommended. The backing pump then should be operated for short times only. The maximum admissible backing pressure, however, must never be exceeded.

The size of the ballast volume depends on the total quantity of gas to be pumped per unit of time. If this rate is very low, the rule of thumb indicates that 0.5 l of ballast volume allows 1 min of pumping time with the backing pump isolated.

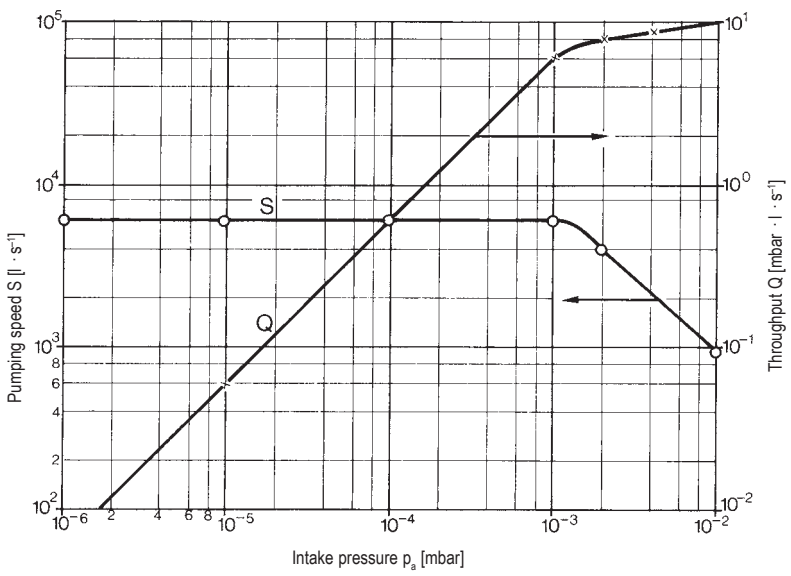
For finding the most adequate size of backing pump, a graphical method may be used in many cases. In this case the starting point is the pumping speed characteristic of the pumps according to equation 2.41.

The pumping speed characteristic of a pump is easily derived from the measured pumping speed (volume flow rate) characteristic of the pump as shown for a 6000 l/s diffusion pump (see curve S in Fig. 2.76). To arrive at the throughput characteristic (curve Q in Fig. 2.76), one must multiply each ordinate value of S by its corresponding pA value and plotted against this value. If it is assumed that the inlet pressure of the diffusion pump does not exceed 10^{-2} mbar, the maximum throughput is 9.5 mbar · l/s

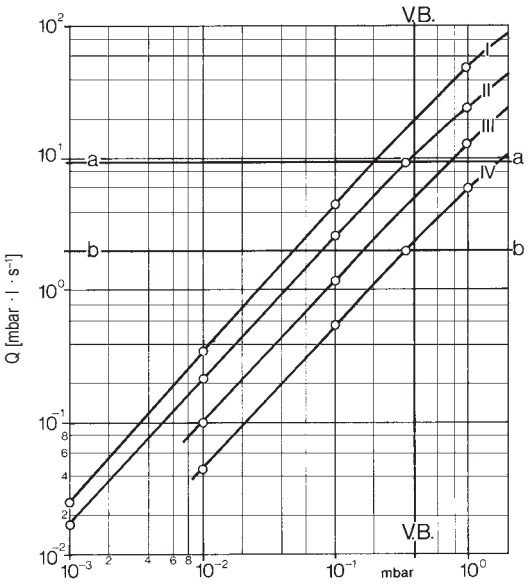
Hence, the size of the backing pump must be such that this throughput can be handled by the pump at an intake pressure (of the backing pump) that is equal to or preferably lower than the maximum permissible backing pressure of the diffusion pump; that is, $4 \cdot 10^{-1}$ mbar for the 6000 l/s diffusion pump.

After accounting for the pumping speed characteristics of commercially available two-stage rotary plunger pumps, the throughput characteristic for each pump is calculated in a manner similar to that used to find the Q curve for the diffusion pump in Fig. 2.76 a. The result is the group of Q curves numbered 1 – 4 in Fig. 2.76 b, whereby four 2-stage rotary-plunger pumps were considered, whose nominal speeds were 200, 100, 50, and 25 m^3/h, respectively. The critical backing pressure of the 6000 l/s diffusion pump is marked as V.B. ($p = 4 \cdot 10^{-1}$ mbar). Now the maximum throughput Q = 9.5 mbar · l/s is shown as horizontal line a. This line intersects the four throughput curves. Counting from right to left, the first point of intersection that corresponds to an intake pressure below the critical backing pressure

Vacuum generation



a) Pumping speed characteristic of a 6000 l/s diffusion pump



b) Series of throughput curves for two-stage rotary plunger pumps (V.B. = Critical forevacuum pressure)

Fig. 2.76 Diagram for graphically determining a suitable backing pump

of $4 \cdot 10^{-1}$ mbar is made with throughput characteristic 2. This corresponds to the two-stage rotary plunger pump with a nominal pumping speed of 100 m^3/h. Therefore, this pump is the correct backing pump for the 6000 l/s diffusion pump under the preceding assumption.

However, if the pumping process is such that the maximum throughput of 9.5 mbar · l/s is unlikely, a smaller backing pump can, of course, be used. This is self-explanatory, for example, from line b in Fig. 2.76 b, which corresponds to a maximum throughput of only 2 mbar l/s. In this case a 25 m^3/h two-stage rotary-plunger pump would be sufficient.

2.3.3 Determination of pump-down time from nomograms

In practice, for instance, when estimating the cost of a planned vacuum plant, calculation of the pump-down time from the effective pumping speed S_{eff}, the required pressure p, and the chamber volume V by formulas presented would be too troublesome and time-consuming. Nomograms are very helpful here. By using the nomogram in Fig. 9.7 in Section 9, one can quickly estimate the pump-down time for vacuum plants evacuated with rotary pumps, if the pumping speed of the pump concerned is fairly constant through the pressure region involved. By studying the examples presented, one can easily understand the application of the nomogram.

The pump-down times of rotary vane and rotary piston pumps, insofar as the pumping speed of the pump concerned is constant down to the required pressure, can be determined by reference to example 1.

In general, Roots pumps do not have constant pumping speeds in the working region involved. For the evaluation of the pump-down time, it usually suffices to assume the mean pumping speed. Examples 2 and 3 of the nomogram show, in this context, that for Roots pumps, the compression ratio K refers not to the atmospheric pressure (1013 mbar), but to the pressure at which the Roots pump is switched on.

In the medium vacuum region, the gas evolution or the leak rate becomes significantly evident. From the nomogram 9.10 in Section 9, the corresponding calculations of the pump-down time in this vacuum region can be approximated.

In many applications it is expedient to relate the attainable pressures at any given time to the pump-down time. This is easily possible with reference to the nomogram 9.7 in Section 9.

As a first example the pump-down characteristic – that is, the relationship pressure p (denoted as desired pressure p_{end}) versus pumping time t_p – is derived from the nomogram for evacuating a vessel of 5 m^3 volume by the single-stage rotary plunger pump E 250 with an effective pumping speed of S_{eff} = 250 m^3/h and an ultimate pressure $p_{end,p} = 3 \cdot 10^{-1}$ mbar when operated with a gas ballast and at $p_{end,p} = 3 \cdot 10^{-2}$ mbar without a gas ballast. The time constant $\tau = V / S_{eff}$ (see equation 2.36) is the same in both cases and amounts as per nomogram 9.7 to about 70 s (column 3). For any given value of $p_{end} > p_{end,p}$ the straight line connecting the "70 s point" on column 3 with the $(p_{end} - p_{end,p})$ value on the **right-hand** scale of column 5 gives the corresponding t_p value. The results of this procedure are shown as curves a and b in Fig. 2.77.

It is somewhat more tedious to determine the (p_{end}, t_p) relationship for a combination of pumps. The second example discussed in the following deals with evacuating a vessel of 5 m^3 volume by the pump combination Roots pump WA 1001 and the backing pump E 250 (as in the preceding example). Pumping starts with the E 250 pump operated without gas ballast alone, until the Roots pump is switched on at the pressure of 10 mbar. As the pumping speed characteristic of the combination WA 1001/ E 250 – in contrast to the characteristic of the E 250 – is no longer a horizontal straight line over the best part of the pressure range (compare this to the

corresponding course of the characteristic for the combination WA 2001 / E 250 in Fig. 2.19), one introduces, as an approximation, average values of S_{eff}, related to defined pressure ranges. In the case of the WA 1001/ E 250 combination the following average figures apply:

S_{eff} = 800 m³/h in the range 10 – 1 mbar,

S_{eff} = 900 m³/h in the range 1 mbar to $5 \cdot 10^{-2}$ mbar,

S_{eff} = 500 m³/h in the range $5 \cdot 10^{-2}$ to $5 \cdot 10^{-3}$ mbar

The ultimate pressure of the combination WA 1001 / E 250 is: $P_{end,p} = 3 \cdot 10^{-3}$ mbar. From these figures the corresponding time constants in the nomogram can be determined; from there, the pump-down time tp can be found by calculating the pressure reduction R on the left side of column 5. The result is curve c in Fig. 2.77.

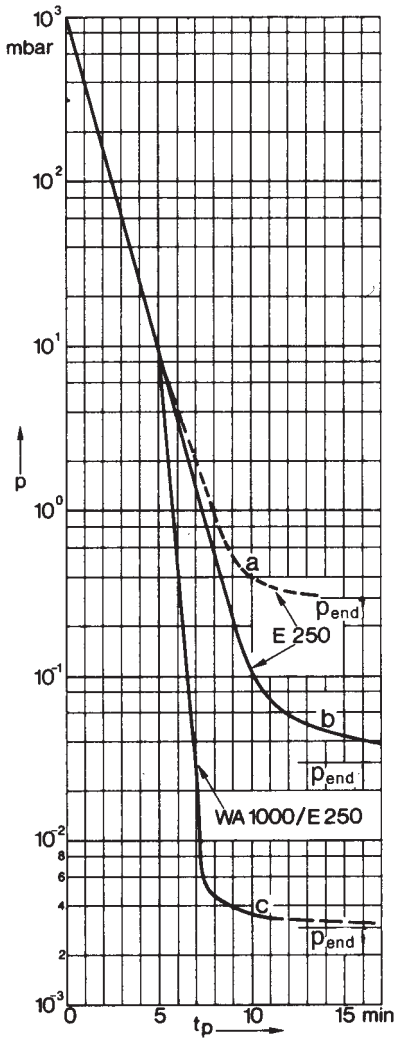


Fig. 2.77 Pumpdown time, t_p, of a 5 m³ vessel using a rotary plunger pump E 250 having a nominal pumping speed of 250 m³/h with (a) and without (b) gas ballast, as well as Roots/rotary plunger pump combination WA 1001 / E250 for a cut-in pressure of 10 mbar for the WA 1001 (e).

Computer aided calculations at LEYBOLD

Of course calculations for our industrial systems are performed by computer programs. These require high performance computers and are thus usually not available for simple initial calculations.

2.3.4 Evacuation of a chamber where gases and vapors are evolved

The preceding observations about the pump-down time are significantly altered if vapors and gases arise during the evacuation process. With bake-out processes particularly, large quantities of vapor can arise when the surfaces of the chamber are cleared of contamination. The resulting necessary pump-down time depends on very different parameters. Increased heating of the chamber walls is accompanied by increased desorption of gases and vapors from the walls. However, because the higher temperatures result in an accelerated escape of gases and vapors from the walls, the rate at which they can be removed from the chamber is also increased.

The magnitude of the allowable temperature for the bake-out process in question will, indeed, be determined essentially by the material in the chamber. Precise pump-down times can then be estimated by calculation only if the quantity of the evolving and pumped vapors is known. However, since this is seldom the case except with drying processes, a quantitative consideration of this question is abandoned within the scope of this publication.

2.3.5 Selection of pumps for drying processes

Fundamentally, we must distinguish between short-term drying and drying processes that can require several hours or even days. Independently of the duration of drying, all drying processes proceed approximately as in Section 2.24

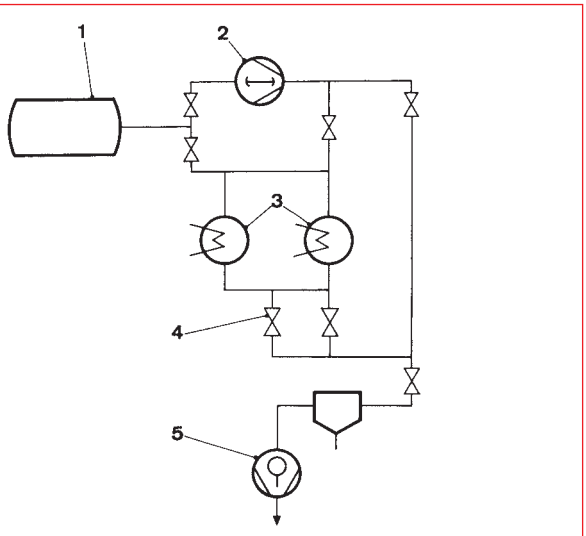
As an example of an application, the drying of salt (short-term drying) is described, this being an already well-proven drying process.

Drying of salt

First, 400 kg of finely divided salt with a water content of about 8 % by mass is to be dried in the shortest possible time (about 1 h) until the water content is less than 1 % by mass. The expected water evolution amounts to about 28 kg. The salt in the chamber is continuously agitated during the drying process and heated to about 80 °C. The vacuum system is schematically drawn in Fig. 2.78.

During the first quarter of drying time far more than half the quantity of water vapor is evolved. Then the condenser is the actual main pump. Because of the high water vapor temperature and, at the beginning of the drying, the very high water vapor pressure, the condensation efficiency of the condenser is significantly increased. In Fig. 2.78 it is understood that two parallel condensers each of 1 m² condensation surface can together condense about 15 l of water at an inlet pressure of 100 mbar in 15 min. However, during this initial process, it must be ensured that the water vapor pressure at the inlet port of the rotary piston pump does not exceed 60

Vacuum generation



1 Vacuum chamber with salt filling
2 RUVAC WA 501
3 Condensers
4 Throttle valve
5 Rotary plunger pump

Fig. 2.78 Vacuum diagram for drying of salt. Pump combination consisting of Roots pump, condenser and rotary plunger pump for stepwise switching of the pumping process (see text)

mbar (see Section 2.15 for further details). Since the backing pump has only to pump away the small part of the noncondensable gases at this stage, a single-stage rotary piston pump TRIVAC S 65 B will suffice. With increasing process time, the water vapor evolution decreases, as does the water vapor pressure in the condenser. After the water pressure in the chamber falls below 27 mbar, the Roots pump (say, a Roots pump RUVAC WA 501) is switched in. Thereby the water vapor is pumped more rapidly out of the chamber, the pressure increases in the condensers, and their condensation efficiency again increases. The condensers are isolated by a valve when their water vapor reaches its saturation vapor pressure. At this point, there is a water vapor pressure in the chamber of only about 4 mbar, and pumping is accomplished by the Roots pump with a gas ballast backing pump until the water vapor pressure reaches about 0.65 mbar. From experience it can be assumed that the salt has now reached the desired degree of dryness.

Drying of paper

If the pumps are to be of the correct size for a longer process run, it is expedient to break down the process run into characteristic sections. As an example, paper drying is explained in the following where the paper has an initial moisture content of 8 %, and the vessel has the volume V.

1. Evacuation

The backing pump must be suitably rated with regard to the volume of the vessel and the desired pump-down time. This pump-down time is arranged according to the desired process duration: if the process is to be finished after 12 – 15 h, the pump-down time should not last longer than 1 h. The size of the backing pump may be easily calculated according to Section 2.3.1.

2. Predrying

During predrying – depending on the pressure region in which the work is carried out – about 75 % of the moisture is drawn off. This predrying should occupy the first third of the drying time. The rate at which predrying proceeds depends almost exclusively on the sufficiency of the heat supply. For predrying 1 ton of paper in 5 h, 60 kg of water must be evaporated; that is, an energy expenditure of about 40 kWh is needed to evaporate water. Since the paper must be heated to a temperature of about 120 °C at the same time, an average of about 20 kW must be provided. The mean vapor evolution per hour amounts to 12 kg. Therefore, a condenser with a capacity of 15 kg/h should be sufficient. If the paper is sufficiently preheated (perhaps by air-circulation drying) before evacuation, in the first hour of drying, double vapor evolution must be anticipated.

3. Main drying

If, in the second stage, the pressure in a further 5 h is to fall from 20 to about 5.3 mbar and 75 % of the total moisture (i.e., 19 % of the total moisture of 15 kg) is to be drawn off, the pump must, according to equations (2.37) and (2.38), have a pumping speed of

$$S_{eff} = \frac{V \cdot \Delta p}{t \cdot p}$$

According to equation 1.7, 15 kg of water vapor corresponds at 15 °C to a quantity of water vapor of

$$V \cdot \Delta p = \frac{m \cdot R \cdot T}{M} = \frac{15 \cdot 83.14 \cdot 288}{18} \approx$$

$$\approx 20000 \text{ mbar} \cdot \text{m}^3 \quad \text{subsequently}$$

$$S_{eff} = \frac{20000}{5 \cdot 5.3} = 750 \text{ m}^3/\text{h}$$

Hence the Roots pump RUVAC WA 1001 would be the suitable pump. The permissible remaining moisture in the product determines the attainable ultimate pressure. The relationship between the ultimate pressure and the remaining moisture is fixed for every product but different from product to product. LEYBOLD has many years of experience to its record regarding applications in this area. Assume that a 0.1 % residual moisture content is required, for which the necessary ultimate pressure is $6 \cdot 10^{-2}$ mbar. During the last 5 h the remaining 6 % of the moisture content, or 5 kg of water, is removed. At a mean pressure of about 0.65 mbar, 2000 m^3/h of vapor is evolved. Two possibilities are offered:

a) One continues working with the above-mentioned Roots pump WA 1001. The ultimate total pressure settles at a value according to the water vapor quantity evolving. One waits until a pressure of about $6.5 \cdot 10^{-2}$ mbar is reached, which naturally takes a longer time.

b From the beginning, a somewhat larger Roots pump is chosen (e.g., the RUVAC WA 2001 with a pumping speed of 2000 m^3/h is suitable). For larger quantities of paper (5000 kg, for example) such a pumping system will be suitable which at a pumping speed for water vapor of up to 20,000 m^3/h automatically lowers the pressure from 27 to 10^{-2} mbar. The entire time need for drying is significantly reduced when using such pumps.

2.3.6 Flanges and their seals

In general, demountable joints in metallic vacuum components, pumps, valves, tubulations, and so on are provided with flanges. Vacuum components for rough, medium, and high vacuum from LEYBOLD are equipped with the following standardized flange systems:

- **Small flanges (KF)** (quick-action connections to DIN 28 403) of nominal widths **10, 16, 20, 25, 32, 40** and **50** mm. The values 10, 16, 25 and 40 are preferred widths according to the PNEUROP recommendations and the ISO recommendations of the technical committee ISO/TC 112 (see also Section 11). For a complete connection of two identical flanges one clamping ring and one centering ring are required.
- **Clamp flanges (ISO-K)** of nominal widths **65, 100, 160, 250, 320, 400, 500** and **630** mm. Also, these flanges correspond to the nominal widths and construction of the PNEUROP and ISO/TC 112 recommendations. Clamp flanges are joined together by clamps or collar rings. Centering rings or gaskets are needed for sealing.
- B**olted flanges (ISO-F)** for the same nominal widths as above (according to PNEUROP and ISO/TC 112). In special cases bolted flanges having a smaller nominal width are used. Clamp flanges and bolted flanges are in accordance with DIN 28 404.

The nominal width is approximately equal to the free inner diameter of the flange in millimeters; greater deviations are exceptions, so the clamp flange DN 63 has an inner diameter of 70 mm. See also Table XI in Section 9).

High vacuum components are made of aluminum or stainless steel. Stainless steel is slightly more expensive but offers a variety of advantages: lower degassing rate, corrosion resistant, can be degassed at temperatures up to 200 °C, metal seals are possible and stainless steel is much more resistant to scratching compared to aluminum.

Ultrahigh vacuum components are made of stainless steel and have **CF flanges** bakeable to a high temperature. These components, including the flanges, are manufactured in a series production, starting with a nominal width of 16 up to 250 mm. CF flanges are available as fixed flanges or also with rotatable collar flanges. They may be linked with CONFLAT flanges from almost all manufacturers. Copper gaskets are used for sealing purposes.

Basically, the flanges should not be smaller than the connecting tubes and the components that are joined to them. When no aggressive gases and vapors are pumped and the vacuum system is not exposed to a temperature above 80 °C, sealing with NBR (Perbunan) or CR (Neoprene) flange O-rings is satisfactory for work in the rough-, medium-, and high vacuum regions. This is often the case when testing the operation of vacuum systems before they are finally assembled.

All stainless steel flanges may be degassed at temperatures up to 200 °C without impairment. However, then Perbunan sealing material is not suitable as a flange sealant. Rather, **VITILAN®** (a special FPM) sealing rings and also **aluminum seals**, which allow heating processes up to **150 °C** and **200 °C** respectively, should be used. After such degassing, pressures down to 10^{-8} mbar, i.e. down to the UHV range, can be attained in vacuum systems.

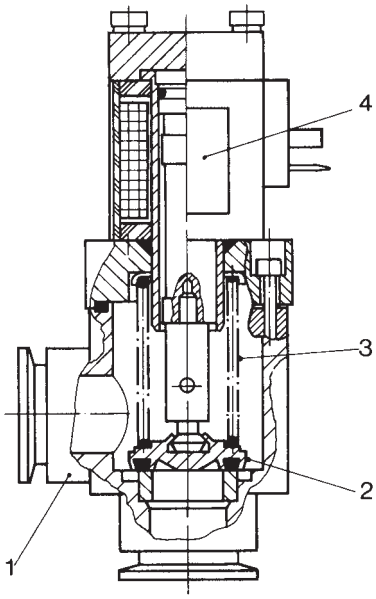
Generating pressures below 10^{-8} mbar requires higher bake-out temperatures. As explained above (see Section 2.2.6) work in the UHV range requires a basically different approach and the use of CF flanges fitted with metallic sealing rings.

2.3.7 Choice of suitable valves

Vacuum technology puts great demands on the functioning and reliability of the valves, which are often needed in large numbers in a plant. The demands are fulfilled only if correct shut-off devices are installed for each application, depending on the method of construction, method of operation, and size. Moreover, in the construction and operation of vacuum plants, factors such as the flow conductance and leak-tightness of valves are of great importance.

Valves are constructed so that they will not throttle pumping speed. Hence, when opened fully, their conductance in the rough and medium vacuum regions equals that of corresponding tube components. For example, the conductance of a right-angle valve will equal the conductance of a bent tube of the same nominal bore and angle. Similarly, the conductance of the valve for molecular flow (i. e., in the high and ultrahigh vacuum regions), is so high that no significant throttling occurs. Actual values for the conductance of various components are given in the catalog.

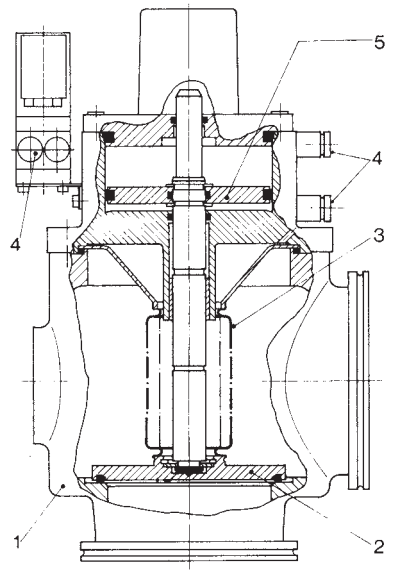
To meet stringent leak-tightness demands, high-quality vacuum valves are designed so that gas molecules adhering to the surface of the valve shaft are not transferred from the outer atmosphere into the vacuum during operation. Such valves are therefore equipped with metal bellows for isolating the valve shaft from the atmosphere, or alternatively, they are fully encapsulated, that is, only static seals exist between atmosphere and vacuum. This group is comprised of all medium and high vacuum valves from LEYBOLD that are operated either manually or electropneumatically (Fig. 2.80) and (Fig. 2.79). The leak rate of these valves is less than 10^{-9} mbar · l/s.



1 Casing
2 Valve disk
3 Compression spring
4 Solenoid coil

Fig. 2.79 Right angle vacuum valve with solenoid actuator

Vacuum generation



1 Casing
2 Valve disk
3 Bellows
4 Compressed air supply
5 Piston

Fig. 2.80 Right angle vacuum valve with electropneumatic actuator

Valves sealed with oil or grease can be used for highly stringent demands. Their leakage rate is also about 10^{-9} mbar · l/s. However, a special case is the pendulum-type gate valve. Despite its grease-covered seal, the leak rate between vacuum and external atmosphere is virtually the same as for bellows-sealed valves because when the valve is in operation the shaft carries out only a rotary motion so that no gas molecules are transferred into the vacuum. Pendulum-type gate valves are not manufactured by LEYBOLD.

For working pressures down to 10^{-7} mbar, valves of standard design suffice because their seals and the housing materials are such that permeation and outgassing are insignificant to the actual process. If pressures down to 10^{-9} mbar are required, baking up to 200 °C is usually necessary, which requires heat resistant sealing materials (e.g., VITILANh) and materials of high mechanical strength, with prepared (inner) surfaces and a low outgassing rate. Such valves are usually made of stainless steel. Flange connections are sealed with aluminum gaskets, so permeation problems of elastomer seals are avoided. In the UHV range these issues are of special significance so that mainly metallic seals must be used. The gas molecules bonded to the surface of the materials have, at pressures below 10^{-9} mbar, a very great influence. They can only be pumped away within a reasonable period of time by simultaneous degassing. Degassing temperatures up to 500 °C required in UHV systems, pose special requirements on the sealing materials and the entire sealing geometry. Gaskets made of gold or copper must be used.

The various applications require valves with different drives, that is, valves that are manually operated, electropneumatically- or magnetically-operated, and motor driven, such as variable-leak valves. The variety is even more enhanced by the various housing designs. In addition to the various materials used, right-angle and straight-through valves are required. Depending on their nominal width and intended application, flanges fitted to valves may be small (KF), clamp (ISO-K), bolted (ISO-F), or UHV (CF).

In addition to the vacuum valves, which perform solely an isolation function (fully open – fully closed position), special valves are needed for special functions. Typical are variable leak valves, which cover the leakage range from 10^{-10} cm³/s (NTP) up to $1.6 \cdot 10^{3}$ cm³/s (NTP). These valves are usually motor driven and suitable for remote control and when they are connected to a pressure gauge, the process pressures can be set and maintained. Other special valves fulfill safety functions, such as rapid, automatic cut-off of diffusion pumps or vacuum systems in the event of a power failure. For example, SECUVAC valves belong to this group. In the event of a power failure, they cut off the vacuum system from the pumping system and vent the forevacuum system. The vacuum system is enabled only after a certain minimum pressure (about 200 mbar) has been attained once the power has been restored.

When aggressive gases or vapors have to be pumped, valves made of stainless steel and sealed with VITILAN® sealant are usually used. For nuclear technology, valves have been developed that are sealed with special elastomer or metal gaskets. We will be pleased to provide further design information for your area of application upon request.

2.3.8 Gas locks and seal-off fittings

In many cases it is desirable not only to be able to seal off gas-filled or evacuated vessels, but also to be in a position to check the pressure or the vacuum in these vessels at some later time and to post-evacuate or supplement or exchange the gas filling.

This can be done quite easily with a seal-off fitting from LEYBOLD which is actuated via a corresponding gas lock. The small flange connection of the evacuated or gas-filled vessel is hermetically sealed off within the tube by a small closure piece which forms the actual valve. The gas lock required for actuation is removed after evacuation or filling with gas. Thus one gas lock will do to actuate any number of seal-off fittings. Shown in Fig. 2.81 is a sectional view of such an arrangement. Gas locks and seal-off fittings are manufactured by LEYBOLD having a nominal width of DN 16 KF, DN 25 KF and DN 40 KF. They are made of stainless steel. The leak rate of the seal-off fittings is less than $1 \cdot 10^{-9}$ mbar l/s. They can sustain overpressures up to 2.5 bar, are temperature resistant up to 150 °C and may be protected against dirt by a standard blank flange.

Typical application examples are double-walled vessels with an insulating vacuum, like Dewar vessels, liquid gas vessels (tanks) or long distance energy pipelines and many more. They are also used for evacuation or post-evacuation of reference and support vacua in scientific instruments seal-off fittings with gas locks are often used. Previously it was necessary to have a pump permanently connected in order to post-evacuate as required. Through the use of gas locks with seal-off fittings a vacuum-tight seal is provided for the vessel and the pump is only required from time to time for checking or post-evacuation.

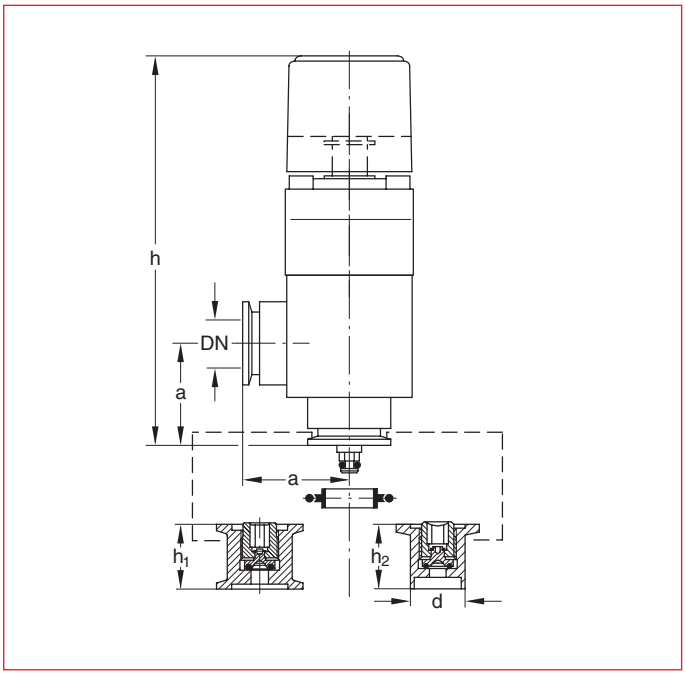


Fig. 2.81 Gas lock with centering ring and seal-off fitting, sectional view

Vacuum measurement

3. Vacuum measurement, monitoring, control and regulation

The pressures measured in vacuum technology today cover a range from 1013 mbar to 10^{-12} mbar, i.e. over 15 orders of magnitude. The enormous dynamics involved here can be shown through an analogy analysis of vacuum pressure measurement and length measurement, as depicted in Table 3.1.

Analogy analysis

Determination by means of	Absolute pressure	Length
empirical world of human beings	1 bar	1 m
simple measuring methods	> 1 mbar	> 1 mm
mechanical measuring methods	> 10^{-3} mbar	> 1 mm
indirect methods	10^{-9} mbar	≈ 1/100 atom∅
extreme indirect methods	10^{-12} mbar	≈ 0.18 electron∅

Table 3.1

Measuring instruments designated as vacuum gauges are used for measurement in this broad pressure range. Since it is impossible for physical reasons to build a vacuum gauge which can carry out quantitative measurements in the entire vacuum range, a series of vacuum gauges is available, each of which has a characteristic measuring range that usually extends over several orders of magnitude (see Fig. 9.16a). In order to be able to allocate the largest possible measuring ranges to the individual types of vacuum gauges, one accepts the fact that the measurement uncertainty rises very rapidly, by up to 100 % in some cases, at the upper and lower range limits. This interrelationship is shown in Fig. 3.1 using the example of the VISCOVAC. Therefore, a distinction must be made between the measuring range as stated in the catalogue and the measuring range for "precise" measurement. The measuring ranges of the individual vacuum gauges are limited in the upper and lower range by physical effects.

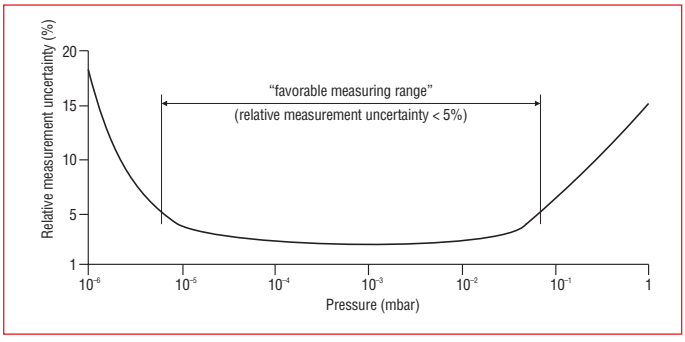


Fig. 3.1 Measurement uncertainty distribution over the measuring range: VISCOVAC

3.1 Fundamentals of low-pressure measurement

Vacuum gauges are devices for measuring gas pressures below atmospheric pressure (DIN 28 400, Part 3, 1992 issue). In many cases the pressure indication depends on the nature of the gas. With compression vacuum gauges it should be noted that if vapors are present, condensation may occur due to the compression, as a result of which the pressure indication is falsified. Compression vacuum gauges measure the sum of the partial pressures of all gas components that do not condense during the measurement procedure. In the case of mechanically compressing pumps, the final partial pressure can be measured in this way (see 1.1). Another way of measuring this pressure, is to freeze out the condensable components in an LN_2 cold trap. Exact measurement of partial pressures of certain gases or vapors is carried out with the aid of partial pressure measuring instruments which operate on the mass spectrometer principle (see section 4).

Dependence of the pressure indication on the type of gas

A distinction must be made between the following vacuum gauges:

1. Instruments that by definition measure the **pressure** as the force **which acts on an area**, the so-called **direct** or **absolute vacuum gauges**. According to the kinetic theory of gases, this force, which the particles exert through their impact on the wall, depends only on the number of gas molecules per unit volume (number density of molecules n) and their temperature, but not on their molar mass. The **reading** of the measuring instrument **is independent of the type of gas**. Such units include liquid-filled vacuum gauges and mechanical vacuum gauges.
2. Instruments with **indirect pressure measurement**. In this case, the pressure is determined as a function of a pressure-dependent (or more accurately, density-dependent) property (thermal conductivity, ionization probability, electrical conductivity) of the gas. These properties are dependent on the molar mass as well as on the pressure. The **pressure reading** of the measuring instrument **depends on the type of gas**.

The scales of these pressure measuring instruments are always based on air or nitrogen as the test gas. For other gases or vapors correction factors, usually based on air or nitrogen, must be given (see Table 3.2). For precise pressure measurement with indirectly measuring vacuum gauges that determine the number density through the application of electrical energy (indirect pressure measurement), it is important to know the gas composition. In practice, the gas composition is known only as a rough approximation. In many cases, however, it is sufficient to know whether light or heavy molecules predominate in the gas mixture whose pressure is to be measured (e.g. hydrogen or pump fluid vapor molecules).

Example: If the pressure of a gas essentially consisting of pump fluid molecules is measured with an ionization vacuum gauge, then the pressure reading (applying to air or N_2), as shown in Table 3.2, is too high by a factor of about 10.

Measurement of pressures in the rough vacuum range can be carried out relatively precisely by means of vacuum gauges with direct pressure measurement. Measurement of lower pressures, on the other hand, is almost always subject to a number of fundamental errors that limit the measuring accuracy right from the start so that it is not comparable at all to the degree

of accuracy usually achieved with measuring instruments. In order to measure pressure in the medium and high vacuum ranges with a measurement uncertainty of less than 50 %, the person conducting the experiment must proceed with extreme care. Pressure measure-ments that need to be accurate to a few percent require great effort and, in general, the deployment of special measuring instruments. This applies particularly to all pressure measurements in the ultrahigh vacuum range ($p < 10^{-7}$ mbar).

To be able to make a meaningful statement about a pressure indicated by a vacuum gauge, one first has to take into account at what location and in what way the measuring system is connected. In all pressure areas where laminar flows prevail ($1013 > p > 10^{-1}$ mbar), note must be taken of pressure gradients caused by pumping. Immediately in front of the pump (as seen from the vessel), a lower pressure is created than in the vessel. Even components having a high conductance may create such a pressure gradient. Finally, the conductance of the connecting line between the vacuum system and the measuring system must not be too small because the line will otherwise be evacuated too slowly in the pressure region of laminar flow so that the indicated pressure is too high.

The situation is more complicated in the case of high and ultrahigh vacuum. According to the specific installation features, an excessively high pressure or, in the case of well-degassed measuring tubes, an excessively low pressure may be recorded due to outgassing of the walls of the vacuum gauge or inadequate degassing of the measuring system. In high and ultrahigh vacuum, pressure equalization between the vacuum system and the measuring tubes may take a long time. If possible, so-called nude gauges are used. The latter are inserted directly in the vacuum system, flange-mounted, without a connecting line or an envelope. Special consideration must always be given to the influence of the measuring process itself on the pressure measurement. For example, in ionization vacuum gauges that work with a hot cathode, gas particles, especially those of the higher hydrocarbons, are thermally broken down. This alters the gas composition. Such effects play a role in connection with pressure measurement in the ultrahigh vacuum range. The same applies to gas clean-up in ionization vacuum gauges, in particular Penning gauges (of the order of 10^{-2} to 10^{-1} l/s). Contamination of the measuring system, interfering electrical and magnetic fields, insulation errors and inadmissibly high ambient temperatures falsify pressure measurement. The consequences of these avoidable errors and the necessary remedies are indicated in the discussion of the individual measuring systems and in summary form in section 8.4.

Selection of vacuum gauges

The desired pressure range is not the only factor considered when selecting a suitable measuring instrument. The operating conditions under which the gauge works also play an important role. If measurements are to be carried out under difficult operating conditions, i.e. if there is a high risk of contamination, vibrations in the tubes cannot be ruled out, air bursts can be expected, etc., then the measuring instrument must be robust. In industrial facilities, Bourdon gauges, diaphragm vacuum gauges, thermal conductivity vacuum gauges, hot cathode ionization vacuum gauges and Penning vacuum gauges are used. Some of these measuring instruments are sensitive to adverse operating conditions. They should and can only be used successfully if the above mentioned sources of errors are excluded as far as possible and the operating instructions are followed.

3.2 Vacuum gauges with pressure reading that is independent of the type of gas

Mechanical vacuum gauges measure the pressure directly by recording the force which the particles (molecules and atoms) in a gas-filled space exert on a surface by virtue of their thermal velocity.

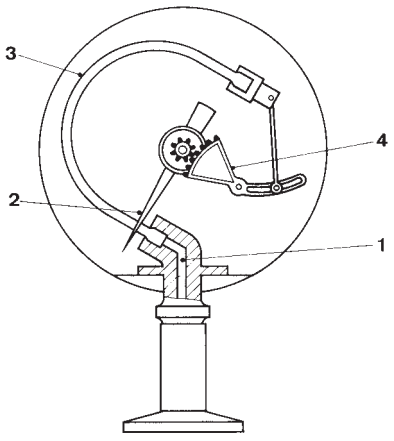
3.2.1 Bourdon vacuum gauges

The interior of a tube bent into a circular arc (so-called Bourdon tube) (3) is connected to the vessel to be evacuated (Fig. 3.2). Through the effect of the external air pressure the end of the tube is deflected to a greater or lesser extent during evacuation and the attached pointer mechanism (4) and (2) is actuated. Since the pressure reading depends on the external atmospheric pressure, it is accurate only to approximately 10 mbar, provided that the change in the ambient atmospheric pressure is not corrected.

3.2.2 Diaphragm vacuum gauges

3.2.2.1 Capsule vacuum gauges

The best-known design of a diaphragm vacuum gauge is a barometer with an aneroid capsule as the measuring system. It contains a hermetically sealed, evacuated, thin-walled diaphragm capsule made of a copper-beryllium alloy. As the pressure drops, the capsule diaphragm expands. This movement is transmitted to a point by a lever system. The **capsule vacuum gauge**, designed according to this principle, indicates the pressure on a linear scale, independent of the external atmospheric pressure.



1 Connecting tube to connection flange
2 Pointer
3 Bourdon tube
4 Lever system

Fig. 3.2 Cross-section of a Bourdon gauge

Vacuum measurement

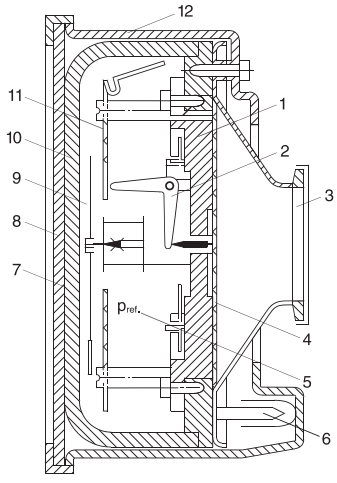
3.2.2.2 DIAVAC diaphragm vacuum gauge

The most accurate pressure reading possible is frequently required for levels below 50 mbar. In this case, a different diaphragm vacuum gauge is more suitable, i.e. the DIAVAC, whose pressure scale is considerably extended between 1 and 100 mbar. The section of the interior in which the lever system (2) of the gauge head is located (see Fig. 3.3) is evacuated to a reference pressure pref of less than 10^{-3} mbar. The closure to the vessel is in the form of a corrugated diaphragm (4) of special steel. As long as the vessel is not evacuated, this diaphragm is pressed firmly against the wall (1). As evacuation increases, the difference between the pressure to be measured px and the reference pressure decreases. The diaphragm bends only slightly at first, but then below 100 mbar to a greater degree. With the DIAVAC the diaphragm deflection is again transmitted to a pointer (9). In particular the measuring range between 1 and 20 mbar is considerably extended so that the pressure can be read quite accurately (to about 0.3 mbar). The sensitivity to vibration of this instrument is somewhat higher than for the capsule vacuum gauge.

3.2.2.3 Precision diaphragm vacuum gauges

A significantly higher measuring accuracy than that of the capsule vacuum gauge and the DIAVAC is achieved by the precision diaphragm vacuum gauge. The design of these vacuum gauges resembles that of capsule vacuum gauges. The scale is linear. The obtainable degree of precision is the maximum possible with present-day state-of-the-art equipment. These instruments permit measurement of 10^{-1} mbar with a full-scale deflection of 20 mbar. The greater degree of precision also means a higher sensitivity to vibration.

Capsule vacuum gauges measure pressure accurately to 10 mbar (due to the linear scale, they are least accurate at the low pressure end of the scale). If only pressures below 30 mbar are to be measured, the DIAVAC



1 Base plate
2 Lever system
3 Connecting flange
4 Diaphragm
5 Reference pressure pref
6 Pinch-off end
7 Mirror sheet
8 Plexiglass sheet
9 Pointer
10 Glass bett
11 Mounting plate
12 Housing

Fig. 3.3 Cross-section of DIAVAC DV 1000 diaphragm vacuum gauge

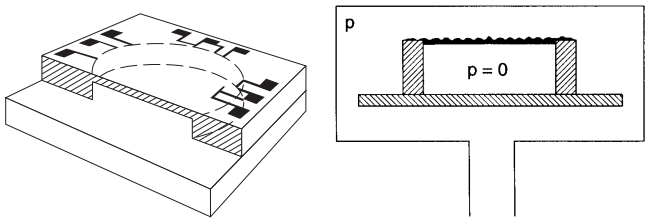


Fig. 3.4 Piezoelectric sensor (basic diagram)

is recommended because its reading (see above) is considerably more accurate. For extremely precise measuring accuracy requirements precision diaphragm vacuum gauges should be used. If low pressures have to be measured accurately and for this reason a measuring range of, for example, up to 20 mbar is selected, higher pressures can no longer be measured since these gauges have a linear scale. All mechanical vacuum gauges are sensitive to vibration to some extent. Small vibrations, such as those that arise in the case of direct connection to a backing pump, are generally not detrimental.

3.2.2.4 Capacitance diaphragm gauges

Deflection of a diaphragm can also be electrically measured as “strain” or as a change in capacitance. In the past, four strain gauges, which change their resistance when the diaphragm is deflected, i.e. under tensile load, were mounted on a metallic diaphragm in a bridge circuit. At LEYBOLD such instruments have been given a special designation, i.e. **MEMBRANOVAC**. Later, silicon diaphragms that contained four such “strain resistances” directly on their surface were used. The electrical arrangement again consisted of a bridge circuit, and a constant current was fed in at two opposite corner points while a linear voltage signal proportional to the pressure was picked up at the two other corner points. Fig. 3.4 illustrates the principle of this arrangement. Such instruments were designated as **PIEZOVAC** units and are still in use in many cases. Today the deflection of the diaphragm is measured as the change in capacitance of a plate capacitor: one electrode is fixed, the other is formed by

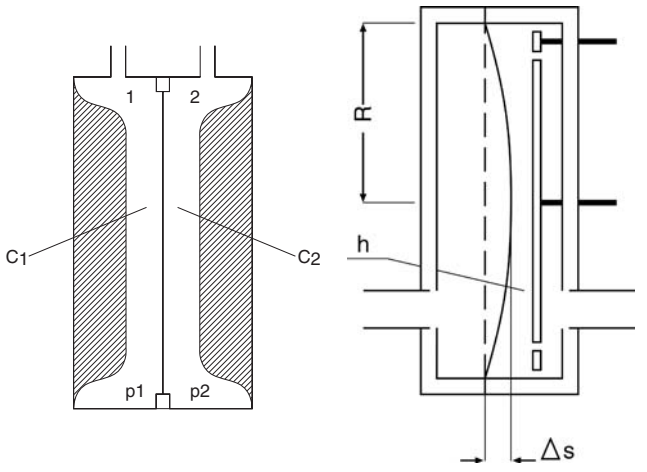
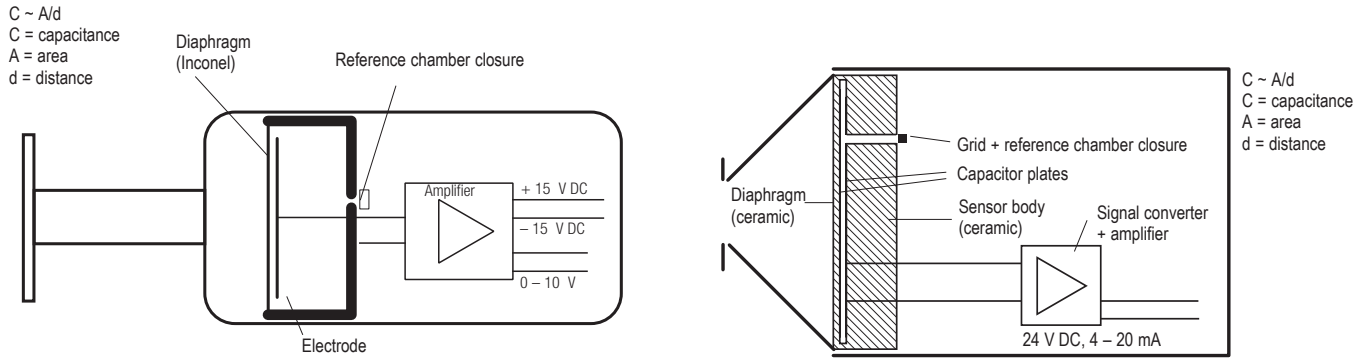


Fig. 3.5 Capacitive sensor (basic diagram)



left: CAPACITRON (Inconel diaphragm)

right: MEMBRANOVAC (aluminum oxide diaphragm)

Fig. 3.6 Capacitive sensors (basic diagram)

the diaphragm. When the diaphragm is deflected, the distance between the electrodes and thus capacitance of the capacitor is altered. Fig. 3.5 illustrates the principle of this arrangement. A distinction is made between sensors with metallic and those with ceramic diaphragms. The structure of the two types is similar and is shown on the basis of two examples in Fig. 3.6. Capacitance diaphragm gauges are used from atmospheric pressure to $1 \cdot 10^{-3}$ mbar (from 10^{-4} mbar the measurement uncertainty rises rapidly). To ensure sufficient deflection of the diaphragms at such low pressures, diaphragms of varying thicknesses are used for the various pressure levels. In each case, the pressure can be measured with the sensors to an accuracy of 3 powers of ten:

1013 to 1 mbar
100 to 10^{-1} mbar
10 to 10^{-2} mbar und
1 to 10^{-3} mbar.

If the pressures to be measured exceed these range limits, it is recommended that a multichannel unit with two or three sensors be used, possibly with automatic channel changeover. The capacitance diaphragm gauge thus represents, for all practical purposes, the only absolute pressure measuring instrument that is independent of the type of gas and designed for pressures under 1 mbar. Today two types of capacitive sensors are available:

1) Sensors DI 200 and DI 2000 with aluminum oxide diaphragms, which are particularly overload-free, with the MEMBRANOVAC DM 11 and DM 12 units.
2) Sensors with Inconel diaphragms CM 1, DM 10, CM 100, CM 1000 with extremely high resolution, with the DM 21 and DM 22 units.

3.2.3 Liquid-filled (mercury) vacuum gauges

3.2.3.1 U-tube vacuum gauges

U-tube vacuum gauges filled with mercury are the simplest and most exact instruments for measuring pressure in the rough vacuum range (1013 to a few mbar). Unfortunately their use in technical plants is limited because of their size and proneness to breakage (see 3.4.1a).

In the evacuated limb of the U-tube vacuum gauge a constant pressure is maintained equal to the vapor pressure of mercury at room temperature (about 10^{-3} mbar). The other limb is connected to the volume in which the pressure is to be measured. From the difference in the levels of the two columns, the pressure to be measured can be determined on the mbar scale provided. The reading is independent of the atmospheric pressure.

3.2.3.2 Compression vacuum gauges (according to McLeod)

The compression vacuum gauge developed by McLeod in 1874 is a very rarely used type of vacuum gauge today. In its refined form the instrument can be used for absolute pressure measurement in the high vacuum range down to 10^{-5} mbar. In the past it was frequently used as a reference instrument for the calibration of medium and sometimes also of high vacuum gauges. For such measurements, however, numerous precautionary rules had to be taken into account before it was possible to assess the measuring accuracy. The pressure is measured by compressing a quantity of gas that initially occupies a large volume into a smaller volume by raising a mercury level. The increased pressure obtained in this manner can be measured in the same way as in a U-tube manometer and from it the original pressure is calculated (see equations below).

According to the type of scale division, a distinction is made between two forms of compression vacuum gauges: those with a linear scale (see Fig. 3.7) and those with a square-law scale (see Fig. 3.8). In the case of the compression vacuum gauges of the McLeod linear-scale type, the ratio of the enclosed residual volume Vc to the total volume V must be known for each height of the mercury level in the measurement capillary; this ratio is shown on the scale provided with the instrument. In the case of compression vacuum gauges with a square-law scale, the total volume and the capillary diameter d must be known.

Nowadays a “shortened” McLeod type compression vacuum gauge according to Kammerer is used to measure the “**partial final pressure**” **of mechanically compressing pumps**. Through the high degree of compres-

Vacuum measurement

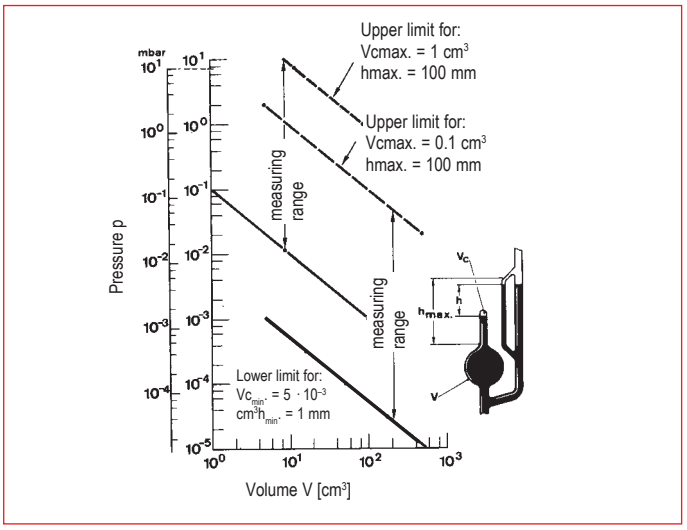


Fig. 3.7 McLeod compression vacuum gauge with linear scale (equation 3.1b)

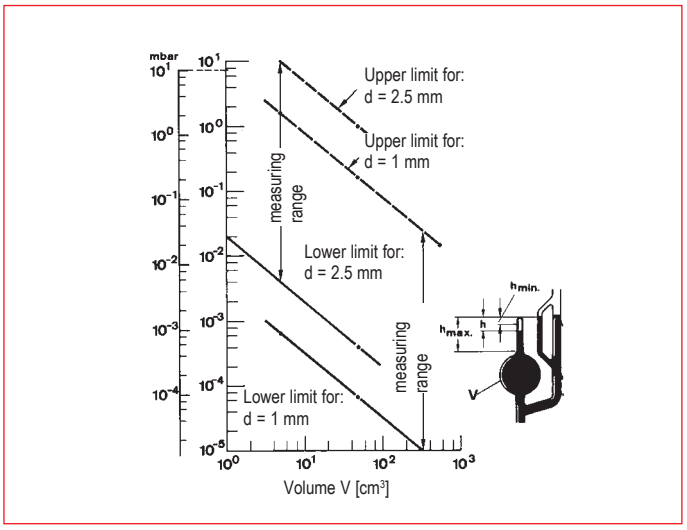


Fig. 3.8 McLeod compression vacuum gauge with square-law scale (equation 3.1f)

sion the condensable gas components (vapors) are discharged as liquid (the volume of the same mass is then smaller by a factor of around 10^5 and can be neglected in the measurement) so that only the pressure of the permanently gaseous components is measured (this is where the expression **permanent gases** comes from).

Principle of measurement with compression vacuum gauges
If h is the difference in the mercury level between the measurement capillary and the reference capillary (measured in mm), then it follows from the Boyle-Mariotte law:

$$p \cdot V = (p + h) \cdot Vc \qquad (3.1)$$

$$p = h \cdot \frac{V_c}{V - V_c} \qquad (3.1a)$$

p measured in mm of mercury (= torr). If V_c << V, then:

$$p = h \cdot \frac{V_c}{V} \qquad (3.1b)$$

V_c and V must be known, h is read off (linear scale).

These relationships remain unchanged if the difference in level is read off a scale with mbar division. The pressure is then obtained in mbar:

$$p = \frac{4}{3} \cdot h \cdot \frac{V_c}{V} \qquad \text{h in mm} \qquad (3.1c)$$

If during measurement the mercury level in the measurement capillary is always set so that the mercury level in the reference capillary corresponds to the upper end of the measurement capillary (see Figs. 3.7 and 3.8), the volume V_c is always given by:

$$V_c = h \cdot \frac{\pi}{4} \cdot d^2 \qquad (3.1d)$$

h ....difference in level, see Fig. 3.5

d ....inside diameter of measurement capillary

If this term is substituted for V_c in equation (3.1b), the result is:

$$p = h^2 \cdot \frac{\pi}{4} \cdot \frac{d^2}{V} \qquad (3.1e)$$

that is, a square-law scale in mm (torr) if d and V are measured in mm or mm^3. If the scale is to be divided into mbar, then the equation is:

$$p = h^2 \cdot \frac{\pi}{3} \cdot \frac{d^2}{V} \qquad (3.1f)$$

where h in mm
d in mm
and V in mm^3

Compression vacuum gauges ensure a reading of the sum of all partial pressures of the permanent gases, provided that no vapors are present that condense during the compression procedure.

The **measuring range** between the top and bottom ends is limited by the maximum and minimum ratios of the capillary volume to the total volume (see Figs. 3.7 and 3.8). The **accuracy of the pressure measurement** depends to a great extent on the reading accuracy. By using a vernier and mirror, pressure measurements with an accuracy of ± 2 % can be achieved. In the low pressure range, where h is very small, this accuracy is no longer attainable, chiefly because small geometric deviations have a very noticeable effect at the closed end of the capillary (systematic error).

The presence of vapors that may condense during compression influences the measurement, often in an indefinite manner. One can easily determine whether vapors having a pressure that is not negligible are present. This can be done by setting different heights h in the measurement capillary under constant pressure while using the linear scale and then calculating p according to equation 3.1b. If no vapors are present, or only those whose pressure is negligible at room temperature (such as mercury), then the same value of p must result for each h.

The scale of compression vacuum gauges can be calculated from the geometric dimensions. This is why they were used in the past by official calibration stations as normal pressure (see equation 3.4.1a).

3.3 Vacuum gauges with gas-dependent pressure reading

This type of vacuum gauge does not measure the pressure directly as an area-related force, but indirectly by means of other physical variables that are proportional to the number density of particles and thus to the pressure. The vacuum gauges with gas-dependent pressure reading include: the decrement gauge (3.3.1), the thermal conductivity vacuum gauge (3.3.2) and the ionization vacuum gauge having different designs (3.3.3).

The instruments consist of the actual sensor (gauge head, sensor) and the control unit required to operate it. The pressure scales or digital displays are usually based on nitrogen pressures; if the true pressure p_T of a gas (or vapor) has to be determined, the indicated pressure p_I must be multiplied by a factor that is characteristic for this gas. These factors differ, depending on the type of instrument, and are either given in tabular form as factors independent of pressure (see Table 3.2) or, if they depend on the pressure, must be determined on the basis of a diagram (see Fig. 3.11).

In general, the following applies:

True pressure p_T = indicated pressure p_I · correction factor

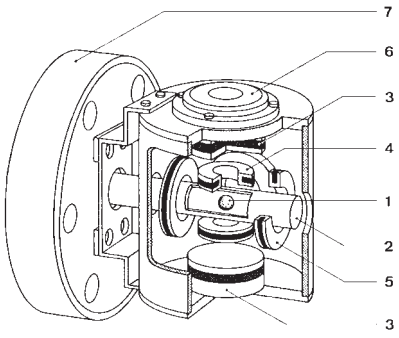
If the pressure is read off a “nitrogen scale” but not corrected, one refers to “nitrogen equivalent” values.

In all electrical vacuum gauges (they include vacuum gauges that are dependent on the type of gas) the increasing use of computers has led to the wish to display the pressure directly on the screen, e.g. to insert it at the appropriate place in a process flow diagram. To be able to use the most standardized computer interfaces possible, so-called **transmitters** (signal converters with standardized current outputs) are built instead of a sensor and display unit (e.g. THERMOVAC transmitter, Penning transmitter, IONIVAC transmitter). Transmitters require a supply voltage (e.g. +24 volts) and deliver a pressure-dependent current signal that is linear over the entire measuring range from 4 to 20 mA or 0 – 10 V. The pressure reading is not provided until after supply of this signal to the computer and processing by the appropriate software and is then displayed directly on the screen.

3.3.1 Spinning rotor gauge (SRG) (VISCOVAC)

Pressure-dependent gas friction at low gas pressures can be utilized to measure pressures in the medium and high vacuum range. In technical instruments of this kind a steel ball that has a diameter of several millimeters and is suspended without contact in a magnetic field (see Fig. 3.9) is used as the measuring element. The ball is set into rotation through an electromagnetic rotating field: after reaching a starting speed (around 425 Hz), the ball is left to itself. The speed then declines at a rate that depends on the prevailing pressure under the influence of the pressure-dependent gas friction. The gas pressure is derived from the relative decline of the speed f (slowing down) using the following equation:

$$-f \cdot \frac{df}{dt} = \frac{10}{\pi} \cdot \frac{p \cdot \sigma}{\bar{c} \cdot r \cdot \rho} \qquad (3.2)$$



1 Ball
2 Measuring tube, closed at one end, welded into connection flange 7
3 Permanent magnets
4 Stabilization coils
5 4 drive coils
6 Bubble level
7 Connection flange

Fig. 3.9 Cross-section of the gauge head of a VISCOVAC VM 212 spinning rotor gauge (SRG)

p = gas pressure
r = radius of the ball
ρ = density of the ball material
$\bar{c}$ = mean speed of the gas particles, dependent on type of gas
σ = coefficient of friction of the ball, independent of the type of gas, nearly 1.

As long as a measurement uncertainty of 3 % is sufficient, which is usually the case, one can apply σ = 1 so that the sensitivity of the spinning rotor gauge (SRG) with rotating steel ball is given by the calculable physical size of the ball, i.e. the product radius x density r · ρ (see equation 3.2). Once a ball has been “calibrated”, it is suitable for use as a “transfer standard”, i.e. as a reference device for calibrating another vacuum gauge through comparison, and is characterized by high long-term stability. Measurements with the VISCOVAC are not limited to measurement of the pressure, however. Other variables involved in the kinetic theory of gases, such as mean free path, monolayer time, particle number density and impingement rate, can also be measured. The instrument permits storage of 10 programs and easy changeover between these programs. The measuring time per slowing-down operation is between 5 seconds for high pressures and about 40 seconds for lower pressures. The measurement sequence of the instrument is controlled fully automatically by a microprocessor so that a new value is displayed after every measurement (slowing-down procedure). The programs additionally enable calculation of a number of statistical variables (arithmetic mean, standard deviation) after a previously specified number of measurements.

While in the case of the kinetic theory of gases with VISCOVAC the counting of particles directly represents the measuring principle (transferring the particle pulses to the rotating ball, which is thus slowed down).

Vacuum measurement

With other electrical measuring methods that are dependent on the type of gas, the particle number density is measured indirectly by means of the amount of heat lost through the particles (thermal conductivity vacuum gauge) or by means of the number of ions formed (ionization vacuum gauge).

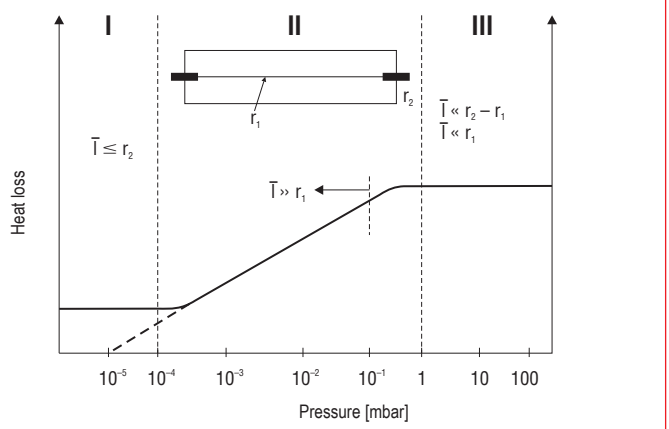
3.3.2 Thermal conductivity vacuum gauges

Classical physics teaches and provides experimental confirmation that the thermal conductivity of a static gas is independent of the pressure at higher pressures (particle number density), p > 1 mbar. At lower pressures, p < 1 mbar, however, the thermal conductivity is pressure-dependent (approximately proportional $1 / \sqrt{M}$). It decreases in the medium vacuum range starting from approx. 1 mbar proportionally to the pressure and reaches a value of zero in the high vacuum range. This pressure dependence is utilized in the thermal conductivity vacuum gauge and enables precise measurement (dependent on the type of gas) of pressures in the medium vacuum range.

The most widespread measuring instrument of this kind is the **Pirani** vacuum gauge. A current-carrying filament with a radius of r_1 heated up to around 100 to 150 °C (Fig. 3.10) gives off the heat generated in it to the gas surrounding it through radiation and thermal conduction (as well as, of course, to the supports at the filament ends). In the rough vacuum range the thermal conduction through gas convection is virtually independent of pressure (see Fig. 3.10). If, however, at a few mbar, the mean free path of the gas is of the same order of magnitude as the filament diameter, this type of heat transfer declines more and more, becoming dependent on the density and thus on the pressure. Below 10^{-3} mbar the mean free path of a gas roughly corresponds to the size of radius r_2 of the measuring tubes. The sensing filament in the gauge head forms a branch of a Wheatstone bridge. In the THERMOTRON **thermal conductivity gauges with variable resistance** which were commonly used in the past, the sensing filament was heated with a constant current. As gas pressure increases, the temperature of the filament decreases because of the greater thermal conduction through the gas so that the bridge becomes out of balance. The bridge current serves as a measure for the gas pressure, which is indicated on a measuring scale. In the THERMOVAC **thermal conductivity gauges with constant resistance** which are almost exclusively built today, the sensing filament is also a branch of a Wheatstone bridge. The heating voltage applied to this bridge is regulated so that the resistance and therefore the temperature of the filament remain constant, regardless of the heat loss. This means that the bridge is always balanced. This mode of regulation involves a time constant of a few milliseconds so that such instruments, in contrast to those with variable resistance, respond very quickly to pressure changes. The voltage applied to the bridge is a measure of the pressure. The measuring voltage is corrected electronically such that an approximately logarithmic scale is obtained over the entire measuring range. Thermal conductivity vacuum gauges with constant resistance have a measuring range from 10^{-4} to 1013 mbar. Due to the very short response time, they are particularly suitable for controlling and pressure monitoring applications (see section 3.5). The measurement uncertainty varies in the different pressure ranges. The maximum error at full-scale deflection is about 1 to 2 %. In the most sensitive range, i.e. between 10^{-3} and 1 mbar, this corresponds to around 10 % of the pressure reading. The measurement uncertainty is significantly greater outside this range.

As in all vacuum gauges dependent on the type of gas, the scales of the indicating instruments and digital displays in the case of thermal conductivity vacuum gauges also apply to nitrogen and air. Within the limits of error, the pressure of gases with similar molecular masses, i.e. O_2, CO and others, can be read off directly. Calibration curves for a series of gases are shown in Fig. 3.11.

An extreme example of the discrepancy between true pressure pT and indicated pressure pI in pressure measurement is the admission of air to a vacuum system with argon from a pressure cylinder to avoid moisture (pumping time). According to Fig. 3.11, one would obtain a p_I reading of only 40 mbar on reaching an “Ar atmospheric pressure” p_T with a THERMOVAC



I Thermal dissipation due to radiation and conduction in the metallic ends
II Thermal dissipation due to the gas, pressure-dependent
III Thermal dissipation due to radiation and convection

Fig. 3.10 Dependence of the amount heat dissipated by a heated filament (radius r_1) in a tube (radius r_2) at a constant temperature difference on the gas pressure (schematic diagram).

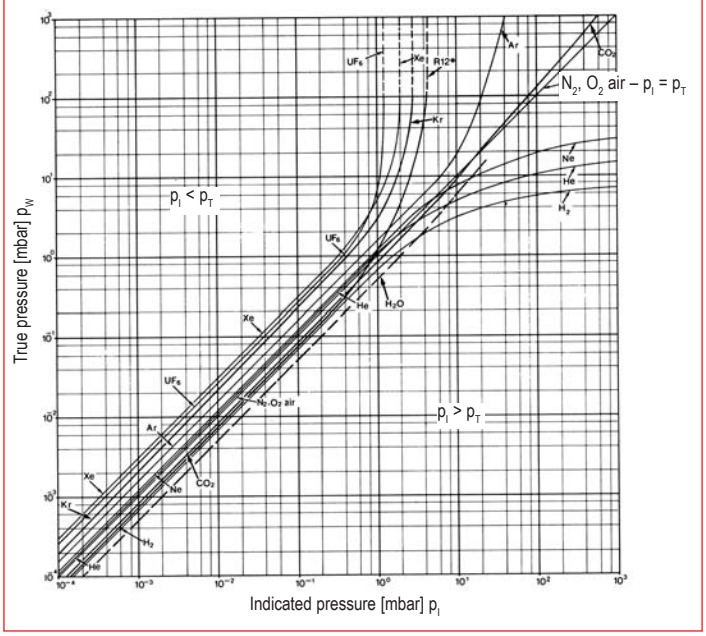


Fig. 3.11 Calibration curves of THERMOVAC gauges for various gases, based on nitrogen equivalent reading

as a pressure measuring instrument. Argon might escape from the vessel (cover opens, bell jar rises). For such and similar applications, pressure switches or vacuum gauges that are independent of the type of gas must be used (see section 3.2).

3.3.3 Ionization vacuum gauges

Ionization vacuum gauges are the most important instruments for measuring gas pressures in the high and ultrahigh vacuum ranges. They measure the pressure in terms of the number density of particles proportional to the pressure. The gas whose pressure is to be measured enters the gauge heads of the instruments and is partially ionized with the help of an electric field. Ionization takes place when electrons are accelerated in the electric field and attain sufficient energy to form positive ions on impact with gas molecules. These ions transmit their charge to a measuring electrode (ion collector) in the system. The ion current, generated in this manner (or, more precisely, the electron current in the feed line of the measuring electrode that is required to neutralize these ions) is a measure of the pressure because the ion yield is proportional to the particle number density and thus to the pressure.

The formation of ions is a consequence of either a discharge at a high electric field strength (so-called cold-cathode or Penning discharge, see 3.3.3.1) or the impact of electrons that are emitted from a hot cathode (see 3.3.3.2).

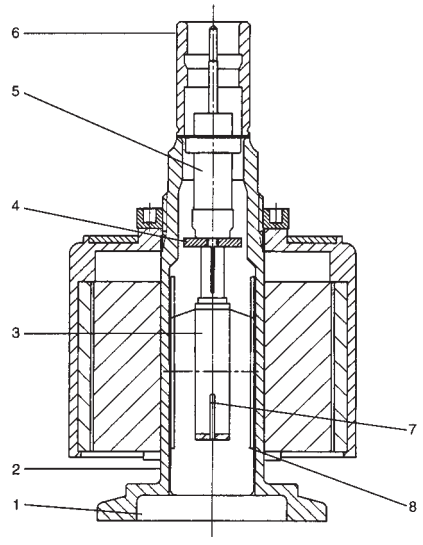
Under otherwise constant conditions, the ion yield and thus the ion current depend on the type of gas since some gases are easier to ionize than others. As all vacuum gauges with a pressure reading that is dependent on the type of gas, ionization vacuum gauges are calibrated with nitrogen as the reference gas (nitrogen equivalent pressure, see 3.3). To obtain the true pressure for gases other than nitrogen, the read-off pressure must be multiplied by the correction factor given in Table 3.2 for the gas concerned. The factors stated in Table 3.2 are assumed to be independent of the pressure, though they depend somewhat on the geometry of the electrode system. Therefore, they are to be regarded as average values for various types of ionization vacuum gauges (see Fig. 3.16).

Given the presence of predominantly (type of gas)	Correction factor based on N_2 (nitrogen = 1)
He	6.9
Ne	4.35
Ar	0.83
Kr	0.59
Xe	0.33
Hg	0.303
H_2	2.4
CO	0.92
CO_2	0.69
CH_4	0.8
higher hydrocarbons	0.1 – 0.4

Table 3.2 Correction factors

3.3.3.1 Cold-cathode ionization vacuum gauges (Penning vacuum gauges)

Ionization vacuum gauges which operate with cold discharge are called cold-cathode- or Penning vacuum gauges. The discharge process in a measuring tube is, in principle, the same as in the electrode system of a sputter ion pump (see section 2.1.8.3). A common feature of all types of cold-cathode ionization vacuum gauges is that they contain just two unheated electrodes, a cathode and an anode, between which a so-called cold discharge is initiated and maintained by means of a d.c. voltage (of around 2 kV) so that the discharge continues at very low pressures. This is achieved by using a magnetic field to make the paths of the electrons long enough so that the rate of their collision with gas molecules is sufficiently large to form the number of charge carriers required to maintain the discharge. The magnetic field (see Fig. 3.12) is arranged such that the magnetic field lines of force cross the electric field lines. In this way the electrons are confined to a spiral path. The positive and negative charge carriers produced by collision move to the corresponding electrodes and form the pressure-dependent discharge current, which is indicated on the meter. The reading in mbar depends on the type of gas. The upper limit of the measuring range is given by the fact that above a level of several
10^{-2} mbar the Penning discharge changes to a glow discharge with intense light output in which the current (at constant voltage) depends only to a small extent on the pressure and is therefore not suitable for measurement purposes. In all Penning gauges there is considerably higher gas sorption than in ionization vacuum gauges that operate with a hot cathode. A Penning measuring tube pumps gases similarly to a sputter ion pump ($S \approx 10^{-2}$ l/s). Here again the ions produced in the discharge are accelerated towards the cathode where they are partly retained and partly cause sputtering of the cathode material. The sputtered cathode material forms



1 Small flange DN 25 KF; DN 40 KF
2 Housing
3 Ring anode with ignition pin
4 Ceramic washer
5 Current leadthrough
6 Connecting bush
7 Anode pin
8 Cathode plate

Fig. 3.12 Cross-section of PENNINGVAC PR 35 gauge

a gettering surface film on the walls of the gauge tube. In spite of these disadvantages, which result in a relatively high degree of inaccuracy in the pressure reading (up to around 50 %), the cold-cathode ionization gauge has three very outstanding advantages. First, it is the least expensive of all high vacuum measuring instruments. Second, the measuring system is insensitive to the sudden admission of air and to vibrations; and third, the instrument is easy to operate.

3.3.3.2 Hot-cathode ionization vacuum gauges

Generally speaking, such gauges refer to measuring systems consisting of three electrodes (cathode, anode and ion collector) where the cathode is a hot cathode. Cathodes used to be made of tungsten but are now usually made of oxide-coated iridium (Th_2O_3, Y_2O_3) to reduce the electron output work and make them more resistant to oxygen. Ionization vacuum gauges of this type work with low voltages and without an external magnetic field. The hot cathode is a very high-yield source of electrons. The electrons are accelerated in the electric field (see Fig. 3.13) and receive sufficient energy from the field to ionize the gas in which the electrode system is located. The positive gas ions formed are transported to the ion collector, which is negative with respect to the cathode, and give up their charge there. The ion current thereby generated is a measure of the gas density and thus of the gas pressure. If i^- is the electron current emitted by the hot cathode, the pressure-proportional current i^+ produced in the measuring system is defined by:

$$i^+ = C \cdot i^- \cdot p \text{ und} \qquad (3.3)$$

$$p = \frac{i^+}{i^- \cdot C} \qquad (3.3a)$$

The variable C is the vacuum gauge constant of the measuring system.

For nitrogen this variable is generally around 10 $mbar^{-1}$. With a constant electron current the sensitivity S of a gauge head is defined as the quotient of the ion current and the pressure. For an electron current of 1 mA and C = 10 mbar-1, therefore, the sensitivity S of the gauge head is:

$S = i^+ / p = C \cdot i^- = 10\ mbar^{-1} \cdot 1\ mA = 10\ mbar^{-1} \cdot 10^{-3}\ A = 1 \cdot 10^{-2}\ A/mbar.$

Hot-cathode ionization vacuum gauges also exhibit gas sorption (pumping action), which, however, is considerably smaller than with Penning systems, i.e. approx. 10^{-3} l/s. Essentially this gas sorption takes place on the glass wall of the gauge head and, to a lesser extent, at the ion collector. Here use is made of nude gauges that are easy to operate because an external magnet is not needed. The upper limit of the measuring range of the hot-cathode ionization gauge is around 10^{-2} mbar (with the exception of special designs). It is basically defined by the scatter processes of ions at gas molecules due to the shorter free path at higher pressures (the ions no longer reach the ion collector = lower ion yield). Moreover, uncontrollable glow or arc discharges may form at higher pressures and electrostatic discharges can occur in glass tubes. In these cases the indicated pressure p_I may deviate substantially from the true pressure p_T.

At low pressures the measuring range is limited by two effects: by the X-ray effect and by the ion desorption effect. These effects results in loss of the strict proportionality between the pressure and the ion current and produce a low pressure threshold that apparently cannot be crossed (see Fig. 3.14).

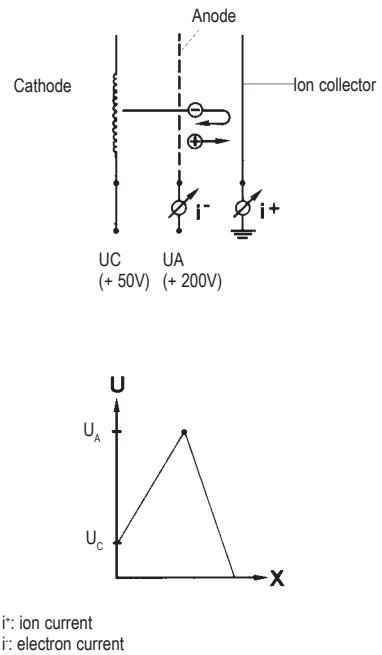


Fig. 3.13 Schematic diagram and potential curve in a hot-cathode ionization vacuum gauge

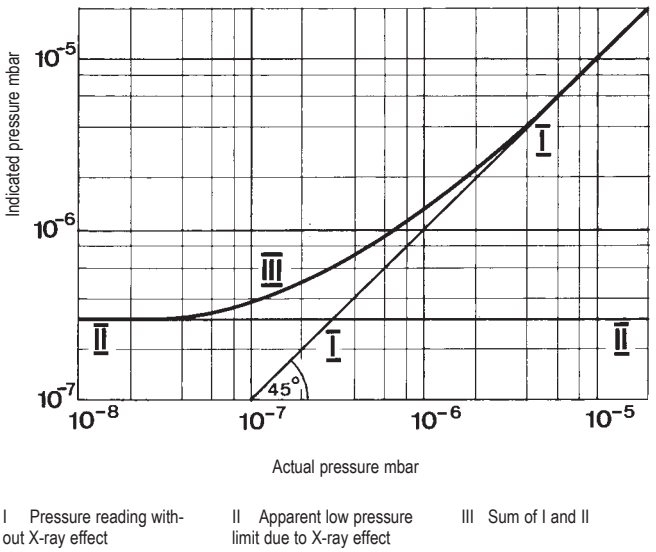


Fig. 3.14 Apparent low pressure limit due to X-ray effect in a normal ionization vacuum gauge

The X-ray effect (see Fig. 3.15)
The electrons emitted from the cathode impinge on the anode, releasing photons (soft X-rays). These photons, in turn, trigger photoelectrons from surfaces they strike. The photoelectrons released from the ion collector flow to the anode, i.e. the ion collector emits an electron current, which is indicated in the same manner as a positive ion current flowing to the ion collector. This photocurrent simulates a pressure. This effect is called the positive X-ray effect, and it depends on the anode voltage as well as on the size of the surface of the ion collector.

Under certain circumstances, however, there is also a negative X-ray effect. Photons which impinge on the wall surrounding the gauge head release photoelectrons there, which again flow towards the anode, and since the anode is a grid structure, they also flow into the space within the anode. If the surrounding wall has the same potential as the ion collector, e.g. ground potential, a portion of the electrons released at the wall can reach the ion collector. This results in the flow of an electron current to the ion collector, i.e. a negative current flows which can compensate the positive ion current. This negative X-ray effect depends on the potential of the outer wall of the gauge head.

The ion desorption effect
Adsorbed gases can be desorbed from a surface by electron impact. For an ionization gauge this means that, if there is a layer of adsorbed gas on the anode, these gases are partly desorbed as ions by the impinging electrons. The ions reach the ion collector and lead to a pressure indication that is initially independent of the pressure but rises as the electron current increases. If such a small electron current is used so that the number of electrons incident at the surface is small compared to the number of adsorbed gas particles, every electron will be able to desorb positive ions. If the electron current is then increased, desorption will initially increase because more electrons impinge on the surface. This finally leads to a reduction in adsorbed gas particles at the surface. The reading falls again and generally reaches values that may be considerably lower than the pressure reading observed with a small electron current. As a consequence of this effect in practice, one must ascertain whether the pressure reading has been influenced by a desorption current. This can be done most simply by temporarily altering the electron current by a factor of 10 or 100. The reading for the larger electron current is the more precise pressure value.

In addition to the conventional ionization gauge, whose electrode structure resembles that of a common triode, there are various ionization vacuum gauge systems (Bayard-Alpert system, Bayard-Alpert system with modulator, extractor system) which more or less suppress the two effects, depending on the design, and are therefore used for measurement in the high and ultrahigh vacuum range. Today the Bayard-Alpert system is usually the standard system.

a) The conventional ionization vacuum gauge
A triode of conventional design (see Fig. 3.16 a) is used as the gauge head, but it is slightly modified so that the outer electrode serves as the ion

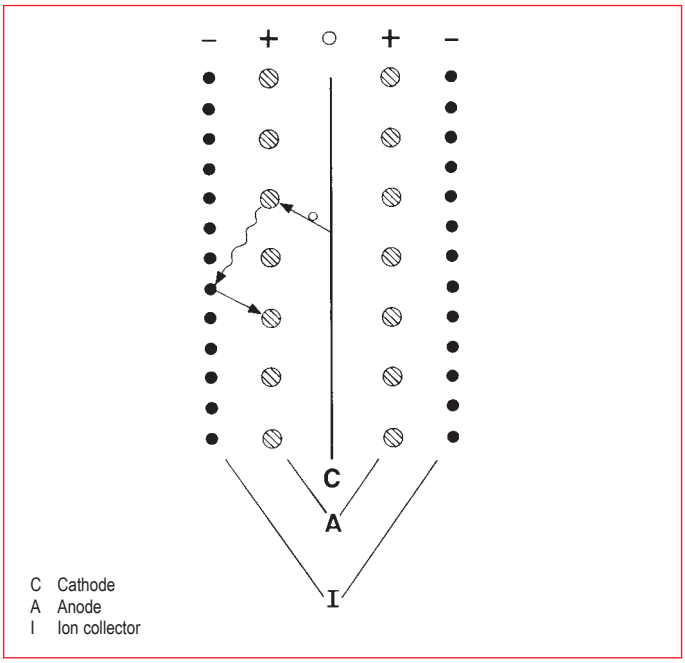


Fig. 3.15 Explanation of the X-ray effect in a conventional ionization gauge. The electrons e^- emitted by the cathode C collide with anode A and trigger a soft X-ray radiation (photons) there. This radiation strikes, in part, the ion collector and generates photoelectrons e_s^- there.

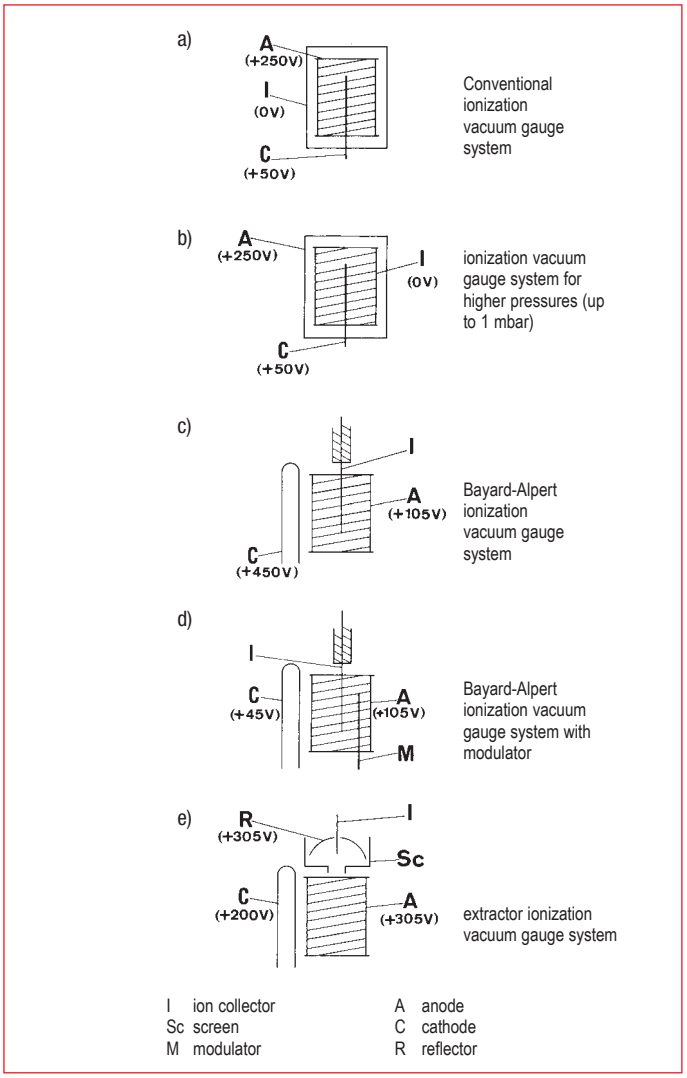


Fig. 3.16 Schematic drawing of the electrode arrangement of various ionization vacuum gauge measuring systems

collector and the grid within it as the anode. With this arrangement the electrons are forced to take very long paths (oscillating around the grid wires of the anode) so that the probability of ionizing collisions and thus the sensitivity of the gauge are relatively high. Because the triode system can generally only be used in high vacuum on account of its strong X-ray effect, the gas sorption (pumping) effect and the gas content of the electrode system have only a slight effect on the pressure measurement.

b) The high-pressure ionization vacuum gauge (up to 1 mbar)
A triode is again used as the electrode system (see Fig. 3.16 b), but this time with an unmodified conventional design. Since the gauge is designed to allow pressure measurements up to 1 mbar, the cathode must be resistant to relatively high oxygen pressure. Therefore, it is designed as a so-called non-burnout cathode, consisting of an yttria-coated iridium ribbon. To obtain a rectilinear characteristic (ion current as a linear function of the pressure) up to a pressure of 1 mbar, a high-ohmic resistor is installed in the anode circuit.

c) Bayard-Alpert ionization vacuum gauge (the standard measuring system used today)
To ensure linearity between the gas pressure and the ion current over as large a pressure range as possible, the X-ray effect must be suppressed as far as possible. In the electrode arrangement developed by Bayard and Alpert, this is achieved by virtue of the fact that the hot cathode is located outside the anode and the ion collector is a thin wire forming the axis of the electrode system (see Fig. 3.16 c). The X-ray effect is reduced by two to three orders of magnitude due to the great reduction in the surface area of the ion collector. When pressures in the ultrahigh vacuum range are measured, the inner surfaces of the gauge head and the connections to the vessel affect the pressure reading. The various effects of adsorption, desorption, dissociation and flow phenomena cannot be dealt with in this context. By using Bayard-Alpert systems as nude gauge systems that are placed directly in the vessel, errors in measurement can be extensively avoided because of the above mentioned effects.

d) Bayard-Alpert ionization vacuum gauge with modulator
The Bayard-Alpert system with modulator (see Fig. 3.16 d), introduced by Redhead, offers pressure measurement in which errors due to X-ray and ion desorption effects can be quantitatively taken into account. In this arrangement there is a second thin wire, the modulator, near the anode in addition to the ion collector inside the anode. If this modulator is set at the anode potential, it does not influence the measurement. If, on the other hand, the same potential is applied to the modulator as that on the ion collector, part of the ion current formed flows to the modulator and the current that flows to the ion collector becomes smaller. The indicated pressure p_I of the ionization gauge with modulator set to the anode potential consists of the portion due to the gas pressure pg and that due to the X-ray effect pg:

$$p_A = p_g + p_\gamma \qquad (3.4)$$

After switching the modulator from the anode potential over to the ion collector potential, the modulated pressure reading p_M is lower than the p_I reading because a portion of the ions now reaches the modulator. Hence:

$$p_M = \alpha \cdot p_g + p\gamma \qquad (3.5)$$

with $\alpha < 1$.

The pg share of the X-ray effect is the same in both cases. After determining the difference between (3.4) and (3.5), we obtain the equation for the gas pressure p_g:

$$p_g = \frac{p_A - p_M}{1-\alpha} \qquad (3.6)$$

α can immediately be determined by experiment at a higher pressure (around 10^{-6} mbar) at which the X-ray effect and thus p_γ are negligible. The pressure corresponding to the two modulator potentials are read off and their ratio is formed. This modulation method has the additional advantage that the ion desorption effect is determined in this way. It permits pressure measurements up to the 10^{-11} mbar range with relatively little effort.

e) Extractor ionization vacuum gauge
Disruptive effects that influence pressure measurement can also be extensively eliminated by means of an ion-optical system first suggested by Redhead. With this extractor system (see Fig. 3.16 e) the ions from the anode cylinder are focused on a very thin and short ion collector. The ion collector is set up in a space, the rear wall of which is formed by a cup-shaped electrode that is maintained at the anode potential so that it cannot be reached by ions emanating from the gas space. Due to the geometry of the system as well as the potential of the of individual electrodes, the disruptive influences through X-ray effects and ion desorption are almost completely excluded without the need of a modulator. The extractor system measures pressures between 10^{-4} and 10^{-12} mbar. Another advantage is that the measuring system is designed as a nude gauge with a diameter of only 35 mm so that it can be installed in small apparatus.

3.4 Adjustment and calibration; DKD, PTB national standards

Definition of terms: Since these terms are often confused in daily usage, a clear definition of them will first be provided:

Adjustment or tuning refers to the correct setting of an instrument. For example, setting 0 and 100 % in THERMOVACs or setting the mass spectrometer to mass 4 in the helium leak detector.

Calibration inspection refers to comparison with a standard in accordance with certain statutory regulations by specially authorized personnel (Bureau of Standards). If the outcome of this regular inspection is positive, an operating permit for the next operation period (e.g. three years) is made visible for outsiders by means of a sticker or lead seal. If the outcome is negative, the instrument is withdrawn from operation.

Calibration refers to comparison with a standard in accordance with certain statutory regulations by specially authorized personnel (calibration facility). The result of this procedure is a calibration certificate which contains the deviations of the readings of the instrument being calibrated from the standard.

Calibration facilities carry out this calibration work. One problem that arises is the question of how good the standards are and where they are calibrated. Such standards are calibrated in calibration facilities of the **German Calibration Service (DKD)**. The German Calibration Service is managed by the Federal Physical-Technical Institute (PTB). Its function is to ensure that measuring and testing equipment used for industrial measurement purposes is subjected to official standards. Calibration of vacuum gauges and test leaks within the framework of the DKD has been assigned to LEYBOLD, as well as other companies, by the PTB. The required calibration pump bench was set up in accordance with DIN 28 418 (see Table 11.1) and then inspected and accepted by the PTB. The standards of the DKD facilities, so-called **transfer standards** (reference vacuum gauges), are calibrated directly by the PTB at regular intervals. Vacuum gauges of all makes are calibrated on an impartial basis by LEYBOLD in Cologne according to customer order. A DKD calibration certificate is issued with all characteristic data on the calibration. The standards of the Federal Physical-Technical Institute are the so-called **national standards**. To be able to guarantee adequate measuring accuracy or as little measurement uncertainty as possible in its calibrations, the PTB largely carries out its measurements through the **application of fundamental methods**. This means, for example, that one attempts to describe the calibration pressures through the measurement of force and area or by thinning the gases in strict accordance with physical laws. The chain of the recalibration of standard instruments carried out once a year at the next higher qualified calibration facility up to the PTB is called “resetting to national standards”. In other countries as well, similar methods are carried out by the national standards institutes as those applied by the Federal Physical-Technical Institute (PTB) in Germany. Fig. 3.17 shows the pressure scale of the PTB. Calibration guidelines are specified in DIN standards (DIN 28 416) and ISO proposals.

3.4.1 Examples of fundamental pressure measurement methods (as standard methods for calibrating vacuum gauges)

a) Measuring pressure with a reference gauge

An example of such an instrument is the U-tube vacuum gauge, with which the measurement of the pressure in the measurement capillary is based on a measurement of the weight over the length of the mercury column.

In the past the McLeod vacuum gauge was also used for calibration purposes. With a precision-made McLeod and carefully executed measurements, taking into account all possible sources of error, pressures down to 10^{-4} mbar can be measured with considerable accuracy by means of such an instrument.

Another reference gauge is the VISCOVAC decrement gauge with rotating ball (see 3.3.1) as well as the capacitance diaphragm gauge (see 3.2.2.4).

b) Generation of a known pressure; static expansion method

On the basis of a certain quantity of gas whose parameters p, V and T are known exactly – p lies within the measuring range of a reference gauge such as a U-tube or McLeod vacuum gauge – a lower pressure within the working range of ionization gauges is reached via expansion in several stages.

If the gas having volume V_1 is expanded to a volume $(V_1 + V_2)$, and from V_2 to $(V_2 + V_3)$, etc., one obtains, after n stages of expansion:

$$p_n = p_1 \cdot \frac{V_1}{V_1 + V_2} \cdot \frac{V_2}{V_2 + V_3} \cdot \ldots \cdot \frac{V_{n-1}}{V_{n-1} + V_n} \qquad (3.7)$$

p_1 = initial pressure measured directly in mbar
p_n = calibration pressure

The volumes here must be known as precisely as possible (see Fig. 3.18) and the temperature has to remain constant. This method requires that the apparatus used be kept very clean and reaches its limit at pressures where the gas quantity can be altered by desorption or adsorption effects beyond

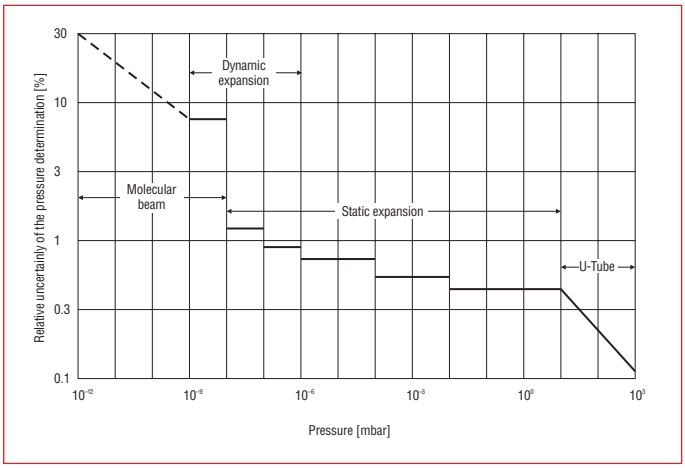


Fig. 3.17 Pressure scale of Federal Physical-Technical Institute (PTB), Berlin, (status as at August 1984) for inert gases, nitrogen and methane

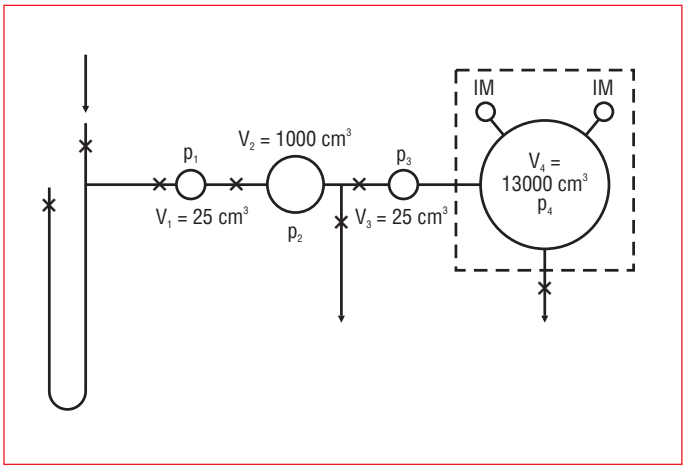
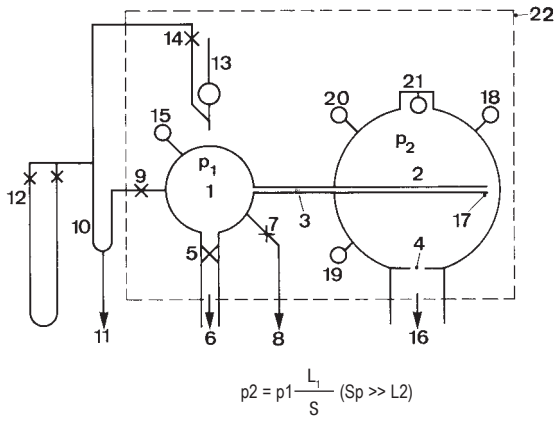


Fig. 3.18 Generation of low pressures through static expansion



$$p2 = p1\frac{L_1}{S} \quad (Sp >> L2)$$

1 Volume 1
2 Volume 2
3 Inlet valve (conductance L1)
4 Aperture with conductance L2
5 Valve
6 to pump system
7 Valve
8 to gas reservoir
9 Valve
10 LN_2 cold trap
11 to pump system
12 U-tube vacuum gauge
13 McLeod vacuum gauge
14 Valve
15 Calibrated ionization gauge tube
16 to pump (pumping speed PS_p)
17 Gas inlet
18 Mass spectrometer
19,20 Gauges to be calibrated
21 Nude gauge to be calibrated
22 Bake-out furnace

Fig. 3.19 Apparatus for calibration according to the dynamic expansion method

the permissible limits of error. According to experience, this lower limit is around $5 \cdot 10^{-7}$ mbar. This method is called the static expansion method because the pressure and volume of the gas at rest are the decisive variables.

c) Dynamic expansion method (see Fig. 3.19)
According to this method, the calibration pressure p is produced by admitting gas at a constant throughput rate Q into a vacuum chamber while gas is simultaneously pumped out of the chamber by a pump unit with a constant pumping speed S. At equilibrium the following applies according to equation 1.10 a:

$$p = Q/S$$

Q is obtained either from the quantity of gas that flows into the calibration chamber from a supply vessel in which constant pressure prevails or from the quantity of gas flowing into the calibration chamber at a measured pressure through a known conductance. The pressure in front of the inlet valve must be high enough so that it can be measured with a reference gauge. The inlet apertures of the valve (small capillaries, sintered bodies) must be so small that the condition $d << \lambda$ is met, i.e. a molecular flow and hence a constant conductance of the inlet valve are obtained (see Section 1.5). The quantity of gas is then defined by $p_1 \cdot L_1$, where p_1 = pressure in front of the inlet valve and L_1 = conductance of the valve. The pumping system consists of a precisely measured aperture with the conductance L_2 in a wall that is as thin as possible (screen conductance) and a pump with a pumping speed of PSp:

$$S = \frac{L_2 \cdot S_p}{L_2 + S_p} = \frac{L_2}{1 + \frac{L_2}{S_p}} \qquad (3.8)$$

and thus

$$p_2 = p_1 \cdot \frac{L_1}{S} = p_1 \cdot \frac{L_1}{L_2} \cdot \left(1 + \frac{L_2}{S_p}\right) \qquad (3.9)$$

This method has the advantage that, after reaching a state of equilibrium, sorption effects can be ignored and this procedure can therefore be used for calibrating gauges at very low pressures.

3.5 Pressure monitoring, control and regulation in vacuum systems

3.5.1 Fundamentals of pressure monitoring and control

In all vacuum processes the pressure in the system must be constantly checked and, if necessary, regulated. Modern plant control additionally requires that all measured values which are important for monitoring a plant are transmitted to central stations, monitoring and control centers and compiled in a clear manner. Pressure changes are frequently recorded over time by recording equipment. This means that additional demands are placed on vacuum gauges:

a) continuous indication of measured values, analog and digital as far as possible

b) clear and convenient reading of the measured values

c) recorder output to connect a recording instrument or control or regulation equipment

d) built-in computer interface (e.g. RS 232)

e) facility for triggering switching operations through built-in trigger points

These demands are generally met by all vacuum gauges that have an electric measured value display, with the exception of Bourdon, precision diaphragm and liquid-filled vacuum gauges. The respective control units are equipped with recorder outputs that supply continuous voltages between 0 and 10 V, depending on the pressure reading on the meter scale, so that the pressure values can be recorded over time by means of a recording instrument. If a pressure switching unit is connected to the recorder output of the gauge, switching operations can be triggered when the values go over or below specified setpoints. The setpoints or switch threshold values for triggering switching operations directly in the gauges are called trigger values. Apart from vacuum gauges, there are diaphragm pressure switches that trigger a switching operation (without display of a measured value) via a contact amplifier when a certain pressure is reached. Valves, for example, can also be controlled through such switching operations.

3.5.2 Automatic protection, monitoring and control of vacuum systems

Protection of a vacuum system against malfunctions is extremely important. In the event of failure, very high material values may be at risk, whether through loss of the entire system or major components of it, due to loss of the batch of material to be processed or due to further production down time. Adequate operational control and protection should therefore be provided for, particularly in the case of large production plants. The individual factors to be taken into account in this connection are best illustrated on the basis of an example: Fig. 3.20 shows the schematic diagram of a high vacuum pump system. The vessel (11) can be evacuated by means of a Roots pump (14) or a diffusion pump (15), both of which operate in conjunction with the backing pump (1). The Roots pump is used in the medium vacuum range and the diffusion pump in the high vacuum range. The valves (3), (8) and (16) are operated electropneumatically. The individual components are actuated from a control panel with pushbuttons. The pump system is to be protected against the following malfunctions:

a) power failure

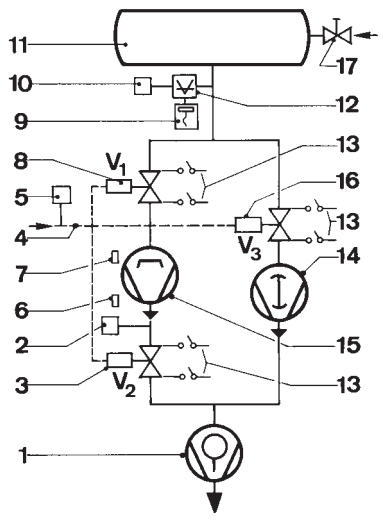
b) drop in pressure in the compressed air network

c) failure of cooling water to the diffusion pump

d) fault in diffusion pump heating system

e) failure of backing pump

f) pressure rise in the vessel above a maximum permissible value



1 Backing pump
2 Backing pressure monitoring device
3 Electropneumatic valve
4 Compressed air connection
5 Pressure monitoring device
6 Temperature monitoring device
7 Cooling water monitoring device
8 Electropneumatic valve
9 Recorder
10 High-vacuum monitoring device
11 Vessel
12 High-vacuum gauge
13 Limit switches
14 Roots pump
15 Diffusion pump
16 Electropneumatic valve
17 Venting valve

Fig. 3.20 Schematic diagram of a high vacuum pump system with optional operation of a Roots pump or a diffusion pump

g) pressure rise above a maximum backing pressure (critical forepressure of the diffusion pump)

The measures to be taken in order to forestall such malfunctions will be discussed in the same order:

a) Measures in the event of power failure: All valves are closed so as to prevent admission of air to the vacuum vessel and protect the diffusion pump against damage.

b) Protection in the event of a drop in pressure in the compressed air network: The compressed air is monitored by a pressure monitoring device (5). If the pressure falls under a specified value, a signal can initially be emitted or the valves can be automatically closed. In this case, a sufficient reserve supply of compressed air is necessary (not shown in Fig. 3.20), which allows all valves to be actuated at least once.

c) Measures in the event of failure of cooling water to the diffusion pump: The cooling water is monitored by a flow or temperature monitoring device (6) and (7). If the flow of cooling water is inadequate, the heater of the diffusion pump is switched off and a signal is given; the valve (8) closes.

d) Protection against failure of the diffusion pump heater: Interruption of the diffusion pump heating system can be monitored by a relay. If the temperature rises above a maximum permissible value, a temperature monitoring device (6) responds. In both cases the valve (8) closes and a signal is given.

e) Protection in the event of backing pump failure: Belt-driven backing pumps must have a centrifugal switch which shuts down the entire system in the event of belt breakage or another malfunction. Monoblock pumps for which the drive is mounted directly on the shaft can be monitored by current relays and the like.

f) Protection against a pressure rise in the vessel above a certain limit value: The high vacuum monitoring device (10) emits a signal when a specified pressure is exceeded.

g) Ensuring the critical forepressure of the diffusion pump: When a certain backing pressure is exceeded, all valves are closed by the backing pressure monitoring device (2), the pumps are switched off and again a signal is given. The position of the valves (3), (8) and (16) is indicated on the control panel by means of limit switches (13). The pressure in the vessel is measured with a high vacuum gauge (12) and recorded with a recorder (9). Protection against operating errors can be provided by interlocking the individual switches so that they can only be actuated in a predetermined sequence. The diffusion pump, for example, may not be switched on when the backing pump is not running or the required backing pressure is not maintained or the cooling water circulation is not functioning.

In principle, it is not a big step from a system protected against all malfunctions to a fully automatic, program-controlled plant, though the complexity of the electrical circuits, of course, increases significantly.

Vacuum measurement

3.5.3 Pressure regulation and control in rough and medium vacuum systems

Control and regulation have the function of giving a physical variable – in this case the pressure in the vacuum system – a certain value. The common feature is the actuator which changes the energy supply to the physical variable and thus the variable itself. Control refers to influencing a system or unit through commands. In this case the actuator and hence the actual value of the physical variable is changed directly with a manipulated variable. Example: Actuation of a valve by means of a pressure-dependent switch. The actual value may change in an undesirable way due to additional external influences. The controlled unit cannot react to the control unit. For this reason control systems are said to have an open operating sequence. In the case of regulation, the actual value of the physical variable is constantly compared to the specified setpoint and regulated if there is any deviation so that it completely approximates the setpoint as far as possible. For all practical purposes regulation always requires control. The main difference is the controller in which the setpoint and the actual value are compared. The totality of all elements involved in the control process forms the control circuit. The terms and characteristic variables for describing control processes are stipulated in DIN 19226.

Generally a distinction is made between discontinuous control (e.g. two-step or three-step control) with specification of a pressure window, within which the pressure may vary, and continuous control (e.g. PID control) with a specified pressure setpoint, which should be maintained as precisely as possible. We have two possible ways of adjusting the pressure in a vacuum system: first, by changing the pumping speed (altering the speed of the pump or throttling by closing a valve); second, through admission of gas (opening a valve). This results in a total of 4 procedures.

Discontinuous pressure regulation

Although continuous regulation undoubtedly represents the more elegant procedure, in many cases two-step or three-step regulation is fully adequate in all vacuum ranges. To specify the pressure window, two or three variable, pressure-dependent switch contacts are necessary. It does not matter here whether the switch contacts are installed in a gauge with display or in a downstream unit or whether it is a pressure switch without display. Fig. 3.21 illustrates the difference between two-step regulation through pumping speed throttling, two-point regulation through gas admission and three-point regulation through a combination of pumping speed throttling and gas admission. Figures 3.22 and 3.23 show the circuit and

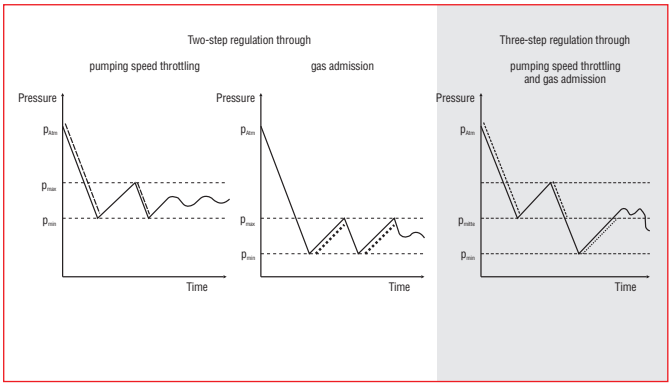
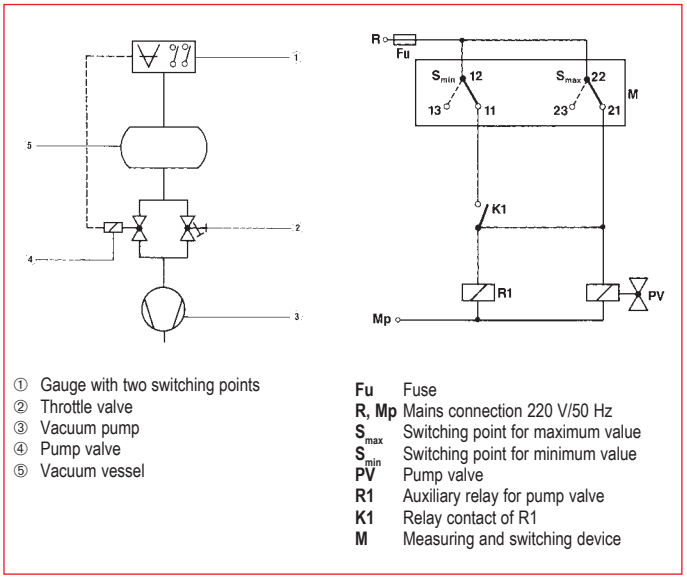


Fig. 3.21 Schematic diagram of two-step and three-step regulation



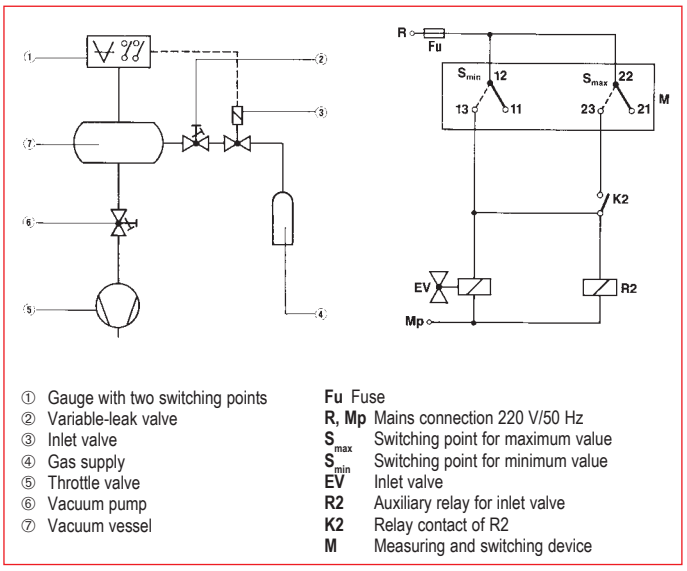
① Gauge with two switching points
② Throttle valve
③ Vacuum pump
④ Pump valve
⑤ Vacuum vessel

Fu Fuse
R, Mp Mains connection 220 V/50 Hz
S_{max} Switching point for maximum value
S_{min} Switching point for minimum value
PV Pump valve
R1 Auxiliary relay for pump valve
K1 Relay contact of R1
M Measuring and switching device

Fig. 3.22 Two-step regulation through pumping speed throttling

structure of the two two-step regulation systems. In the case of two-step regulation through pumping speed throttling (Fig. 3.22), voltage is supplied to pump valve 4, i.e. it is open when the relay contacts are in the release condition. At a level below the upper switching point the valve remains open because of the self-holding function of the auxiliary relay. Only at a level below the lower switching point is the relay latching released. If the pressure subsequently rises, the valve is opened again at the upper switching point.

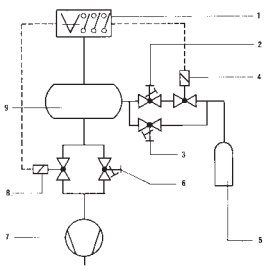
In the case of two-step regulation through gas admission, the inlet valve is initially closed. If the upper pressure switching point is not reached, nothing changes; only when the pressure falls below the lower switching point, do the "make contacts" open the gas inlet valve and actuate the auxiliary relay with self-holding function simultaneously. Return to the idle state with closing of the gas inlet valve is not effected until after the upper switching point is exceeded due to the release of the relay self-holding function.

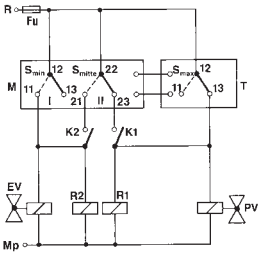


① Gauge with two switching points
② Variable-leak valve
③ Inlet valve
④ Gas supply
⑤ Throttle valve
⑥ Vacuum pump
⑦ Vacuum vessel

Fu Fuse
R, Mp Mains connection 220 V/50 Hz
S_{max} Switching point for maximum value
S_{min} Switching point for minimum value
EV Inlet valve
R2 Auxiliary relay for inlet valve
K2 Relay contact of R2
M Measuring and switching device

Fig. 3.23 Two-step regulation through gas admission





① Gauge with three switching points
② Variable-leak valve
③ Variable-leak valve
④ Inlet valve
⑤ Gas supply
⑥ Throttle valve
⑦ Vacuum pump
⑧ Pump valve
⑨ Vacuum vessel

Fu Fuse
R, Mp Mains connection 220 V/50 Hz
S_{max} Switching point for maximum value
S_{mitte} Switching point for mean value
S_{min} Switching point for minimum value
T TORROSTAT® S 020
PV Pump valve
EV Inlet valve
R1 Auxiliary relay for pump interval
R2 Auxiliary relay for inlet interval
K1 Relay contact of R1
K2 Relay contact of R2
M Measuring and switching device

Fig. 3.24 Three-step regulation system

Fig. 3.24 shows the corresponding three-step regulation system which was created with the two components just described. As the name indicates, two switching points, the lower switching point of the regulation system through pumping speed throttling and the upper switching point of the gas inlet regulation system, were combined.

To avoid the complicated installation with auxiliary relays, many units offer a facility for changing the type of function of the built-in trigger values via software. Initially one can choose between individual switching points (so-called “level triggers”) and interlinked switching points (“interval triggers”). These functions are explained in Fig. 3.25. With interval triggers one can also select the size of the hysteresis and the type of setpoint specification, i.e. either fixed setting in the unit or specification through an external voltage, e.g. from 0 – 10 volts. A three-step regulation system (without auxiliary relay), for example, can be set up with the LEYBOLD MEMBRANOVAC of the A series. Fig. 3.26 shows different units of the new LEYBOLD A series, which, although they function according to different measuring methods, all display a uniform appearance.

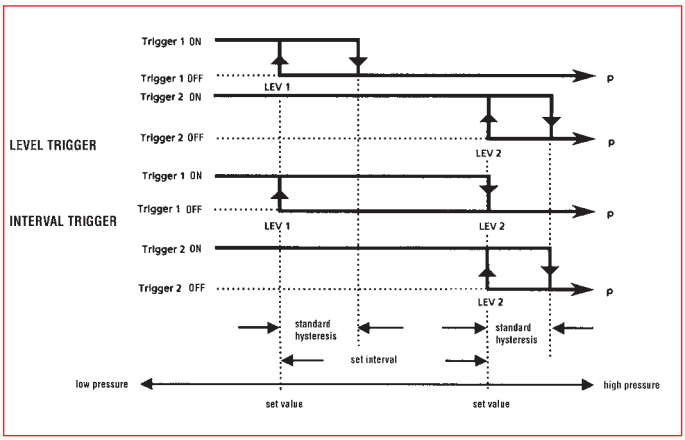


Fig. 3.25 Diagram of level triggers and interval triggers



Fig. 3.26 LEYBOLD-A series, equipment with level and interval triggers

Continuous pressure regulation

We have to make a distinction here between **electric controllers** (e.g. PID controllers) with a proportional valve as actuator and mechanical diaphragm controllers. In a regulation system with electric controllers the coordination between controller and actuator (piezoelectric gas inlet valve, inlet valve with motor drive, butterfly control valve, throttle valve) is difficult because of the very different boundary conditions (volume of the vessel, effective pumping speed at the vessel, pressure control range). Such control circuits tend to vibrate easily when process malfunctions occur. It is virtually impossible to specify generally valid standard values.

Many control problems can be better solved with a diaphragm controller. The function of the **diaphragm controller** (see Fig. 3.27) can be easily derived from that of a diaphragm vacuum gauge: the blunt end of a tube or pipe is either closed off by means of an elastic rubber diaphragm (for reference pressure > process pressure) or released (for reference pressure < process pressure) so that in the latter case, a connection is established between the process side and the vacuum pump. This elegant and more or less “automatic” regulation system has excellent control characteristics (see Fig. 3.28).

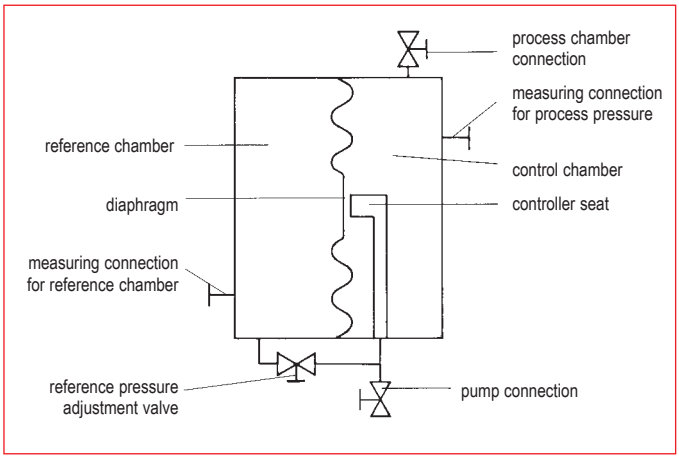
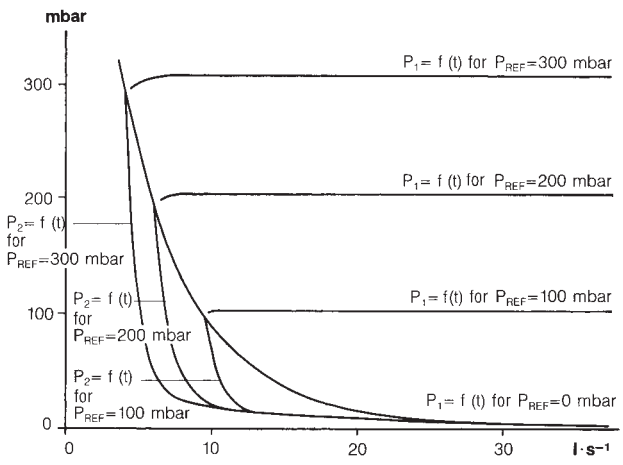


Fig. 3.27 Principle of a diaphragm controller

Vacuum measurement



P_1 = process pressure, P_2 = pressure in pump, P_{ref} = reference pressure

Fig. 3.28 Control characteristics of a diaphragm controller

To achieve higher flow rates, several diaphragm controllers can be connected in parallel. This means that the process chambers and the reference chambers are also connected in parallel. Fig. 3.29 shows such a connection of 3 MR 50 diaphragm controllers.

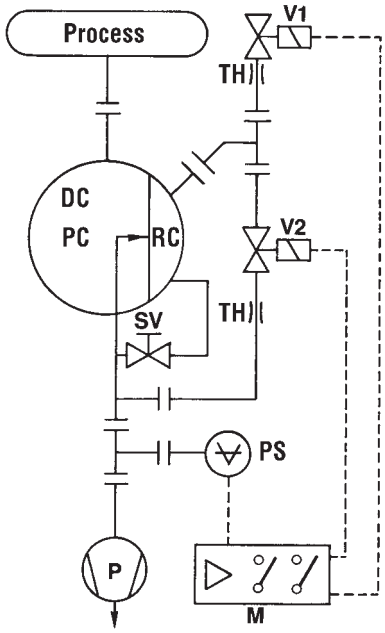
To control a vacuum process, it is frequently necessary to modify the pressure in individual process steps. With a diaphragm controller this can be done either manually or via electric control of the reference pressure.

Electric control of the reference pressure of a diaphragm controller is relatively easy because of the small reference volume that always remains constant. Fig. 3.31 shows such an arrangement on the left as a picture and on the right schematically, see 3.5.5 for application examples with diaphragm controllers.

To be able to change the reference pressure and thus the process pressure towards higher pressures, a gas inlet valve must additionally be installed at the process chamber. This valve is opened by means of a differential pressure switch (not shown in Fig. 3.31) when the desired higher process pressure exceeds the current process pressure by more than the pressure difference set on the differential pressure switch.



Fig. 3.29 Triple connection of diaphragm controllers



DC Diaphragm controller
P Vacuum pump
M Measuring and switching device
PS Pressure sensor
V1 Pump valve
V2 Gas inlet valve
TH Throttle
RC Reference chamber
PC Process chamber
CV Internal reference pressure control valve

Fig. 3.30 Control of vacuum drying processes by regulation of the intake pressure of the vacuum pump according to the water vapor tolerance

3.5.4 Pressure regulation in high and ultrahigh vacuum systems

If the pressure is to be kept constant within certain limits, an equilibrium must be established between the gas admitted to the vacuum vessel and the gas simultaneously removed by the pump with the aid of valves or throttling devices. This is not very difficult in rough and medium vacuum systems because desorption of adsorbed gases from the walls is generally negligible in comparison to the quantity of gas flowing through the system. Pressure regulation can be carried out through gas inlet or pumping speed regulation. However, the use of diaphragm controllers is only possible between atmospheric pressure and about 10 mbar.

In the high and ultrahigh vacuum range, on the other hand, the gas evolution from the vessel walls has a decisive influence on the pressure. Setting of specific pressure values in the high and ultrahigh vacuum range, therefore, is only possible if the gas evolution from the walls is negligible in relation to the controlled admission of gas by means of the pressure-regulating unit. For this reason, pressure regulation in this range is usually effected as gas admission regulation with an electric PID controller. Piezoelectric or servomotor-controlled variable-leak valves are used as actuators. Only bakeable all-metal gas inlet valves should be used for pressure regulation below 10^{-6} mbar.

3.5.5 Examples of applications with diaphragm controllers

1) Regulation of a drying/distillation process, taking into account the maximum water vapor tolerance of a vane type rotary pump

In a drying process it is frequently desirable to carry out drying solely by means of vacuum pumps without inserting condensers. In view of the limited water vapor tolerance of vacuum pumps – approx. 30 mbar as a rule – this would result in condensation of the vapors produced within the vacuum pump, given non-throttled or non-regulated pumping speed. One can avoid this through process-dependent remote control of a diaphragm controller with auxiliary control valves and a measuring and switching device with a pressure sensor at the inlet connection of the vacuum pump if the intake pressure is adapted to the pumps water vapor tolerance through automatic monitoring of the intake pressure of the vacuum pump and by throttling the pumping speed. Fig. 3.30 shows the principle of this arrangement.

Mode of operation: Starting from atmospheric pressure with the process heating switched off, valve V1 is initially open (maximum switching point exceeded) so that atmospheric pressure also prevails in the reference chamber.

The diaphragm controller is therefore closed. When the system is started up, the connecting line between the vacuum pump and pump valve V2 is first evacuated. As soon as the pressure drops below the maximum switching point, valve V1 closes. When the pressure falls below the minimum switching point, valve V2 opens.

In this manner the pressure in the reference chamber is slowly lowered, the throttling of the diaphragm controller is reduced accordingly and thus the process pressure is lowered until the quantity of process gas is greater than the quantity conveyed by the pump so that the minimum switching point is again exceeded. Valve V2 closes again. This interaction repeats itself until the pressure in the process chamber has dropped below the minimum switching point. After that, valve V2 remains open so that the process can be brought down to the required final pressure with a completely open diaphragm controller.

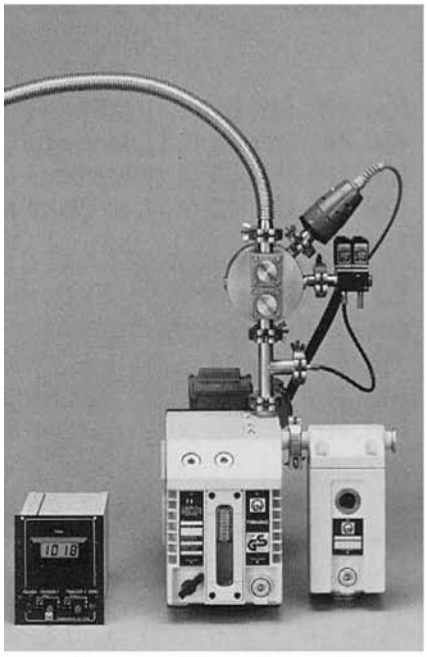
The material to be dried is usually heated to intensify and speed up the drying process. If a certain amount of water vapor is produced, the intake pressure rises above the two switching points. As a result, valve V2 first closes and V1 opens. Through incoming air or protective gas the pressure in the reference chamber is raised and the throughput at the diaphragm controller thus throttled until the intake pressure of the vacuum pump has dropped below the set maximum switching point again. Then valve V1 closes.

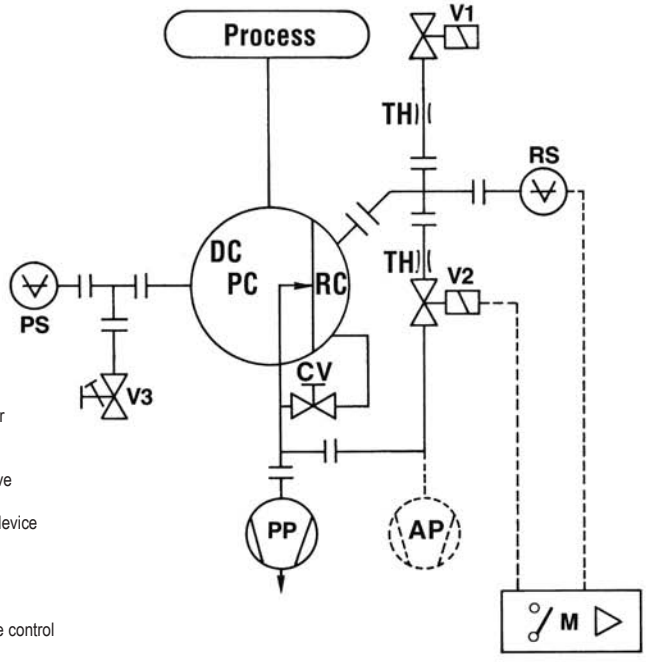
Depending on the quantity of vapor that accumulates, the throughput of the diaphragm controller is set by increasing or decreasing the reference pressure in each case so that the maximum permissible partial water vapor pressure at the inlet connection of the vacuum pump is never exceeded.

As soon as the pressure in the process chamber drops below the set minimum switching point towards the end of the drying process, valve V2 opens and remains open. In this way the unthrottled cross-section of the diaphragm controller is available again for rapid final drying. At the same time the final drying procedure can be monitored by means of the pressure sensor PS.

2) Pressure regulation by means of diaphragm controller with external automatic reference pressure adjustment (see Fig. 3.31)

For automatic vacuum processes with regulated process pressure, presetting of the desired set pressure must often function and be monitored automatically. If a diaphragm controller is used, this can be done by equipping the reference chamber with a measuring and switching device and a control valve block at the reference chamber. The principle of this arrangement is shown in Fig. 3.31.





DC Diaphragm controller
PS Process pressure sensor
RS Reference pressure sensor
V1 Gas inlet valve
V2 Pump valve
V3 Gas inlet variable-leak valve
TH Throttle
M Measuring and switching device
PP Process pump
RC Reference chamber
PC Process chamber
AP Auxiliary pump
CV Internal reference pressure control valve

Fig. 3.31 Diaphragm controller with external automatic reference pressure regulation

Vacuum measurement

Mode of operation: Starting with atmospheric pressure, gas inlet valve V1 is closed at the beginning of the process. Pump valve V2 opens. The process chamber is now evacuated until the set pressure, which is preset at the measuring and switching device, is reached in the process chamber and in the reference chamber. When the pressure falls below the set switching threshold, pump valve V2 closes. As a result, the pressure value attained is “caught” as the reference pressure in the reference chamber (RC) of the diaphragm controller (DC). Now the process pressure is automatically maintained at a constant level according to the set reference pressure by means of the diaphragm controller (DC). If the reference pressure should rise in the course of the process due to a leak, this is automatically detected by the measuring and switching device and corrected by briefly opening pump valve V2. This additional control function enhances the operational reliability and extends the range of application. Correcting the increased reference pressure to the originally set value is of special interest for regulated helium circuits because the pressure rise in the reference chamber (RC) of the diaphragm controller can be compensated for through this arrangement as a consequence of the unavoidable helium permeability of the controller diaphragm of FPM.

To be able to change the reference pressure and thus increase the process pressure to higher pressures, a gas inlet valve must be additionally installed at the process chamber. This valve is opened by means of a differential pressure switch (not shown in Fig. 3.31) when the desired higher process pressure exceeds the current process pressure by more than the pressure differential set at the differential pressure switch.

4. Analysis of gas at low pressures using mass spectrometry

4.1 General

Analyses of gases at low pressures are useful not only when analyzing the residual gases from a vacuum pump, leak testing at a flange connection or for supply lines (compressed air, water) in a vacuum. They are also essential in the broader fields of vacuum technology applications and processes. For example in the analysis of process gases used in applying thin layers of coatings to substrates. The equipment used for qualitative and/or quantitative analyses of gases includes specially developed mass spectrometers with extremely small dimensions which, like any other vacuum gauge, can be connected directly to the vacuum system. Their size distinguishes these measurement instruments from other mass spectrometers such as those used for the chemical analyses of gases. The latter devices are poorly suited, for example, for use as partial pressure measurement units since they are too large, require a long connector line to the vacuum chamber and cannot be baked out with the vacuum chamber itself. The investment for an analytical mass spectrometer would be unjustifiably great since, for example, the requirements as to resolution are far less stringent for partial pressure measurements. Partial pressure is understood to be that pressure exerted by a certain type of gas within a mix of gases. The total of the partial pressures for all the types of gas gives the total pressure. The distinction among the various types of gases is essentially on the basis of their molar masses. The primary purpose of analysis is therefore to register qualitatively the proportions of gas within a system as regards the molar masses and determine quantitatively the amount of the individual types of gases associated with the various atomic numbers.

Partial pressure measurement devices which are in common use comprise the measurement system proper (the sensor) and the control device required for its operation. The sensor contains the ion source, the separation system and the ion trap. The separation of ions differing in masses and charges is often effected by utilizing phenomena which cause the ions to resonate in electrical and magnetic fields.

Initially, the control units were quite clumsy and offered uncountable manipulation options. It was often the case that only physicists were able to handle and use them. With the introduction of PCs the requirements in regard to the control units became ever greater. At first, they were fitted with interfaces for linkage to the computer. Attempts were made later to equip a PC with an additional measurement circuit board for sensor operation. Today's sensors are in fact transmitters equipped with an electrical power supply unit attached direct at the atmosphere side; communication with a PC from that point is via the standard computer ports (RS 232, RS 485). Operating convenience is achieved by the software which runs on the PC.



a: High-performance sensor with Channeltron
b: Compact sensor with Micro-Channelplate
c: High-performance sensor with Faraday cup

Fig. 4.1a TRANSPECTOR sensors

4.2 A historical review

Following Thomson's first attempt in 1897 to determine the ratio of charge to mass e/m for the electron, it was quite some time (into the 1950s) before a large number and variety of analysis systems came into use in vacuum technology. These included the Omegatron, the Topatron and ultimately the quadrupole mass spectrometer proposed by Paul and Steinwedel in 1958, available from INFICON in its standard version as the TRANSPECTOR (see Fig. 4.1). The first uses of mass spectrometry in vacuum-assisted process technology applications presumably date back to Backus' work in the years 1943 / 44. He carried out studies at the Radiographic Laboratories at the University of California. Seeking to separate uranium isotopes, he used a 180° sector field spectrometer after Dempster (1918), which he referred to as a "vacuum analyzer". Even today a similar term, namely the "residual gas analyzer" (RGA), is frequently used in the U.S.A. and the U.K. instead of "mass spectrometer". Today's applications in process monitoring are found above all in the production of semiconductor components.



Fig. 4.1b TRANSPECTOR XPR sensor

4.3 The quadrupole mass spectrometer (TRANSPECTOR)

The ion beam extracted from the electron impact ion source is diverted into a quadrupole separation system containing four rod-shaped electrodes. The cross sections of the four rods form the circle of curvature for a hyperbola so that the surrounding electrical field is nearly hyperbolic. Each of the two opposing rods exhibits equal potential, this being a DC voltage and a superimposed high-frequency AC voltage (Fig. 4.2). The voltages applied induce transverse oscillations in the ions traversing the center, between the rods. The amplitudes of almost all oscillations escalate so that ultimately the ions will make contact with the rods; only in the case of ions with a certain ratio of mass to charge m/e is the resonance condition which allows passage through the system satisfied. Once they have escaped from the separation system the ions move to the ion trap (detector, a Faraday cup) which may also take the form of a secondary electron multiplier pick-up (SEMP).

The length of the sensor and the separation system is about 15 cm. To ensure that the ions can travel unhindered from the ion source to the ion trap, the mean free path length inside the sensor must be considerably greater than 15 cm. For air and nitrogen, the value is about $p \cdot \lambda = 6 \cdot 10^{-3}$ mbar $\cdot$ cm. At $p = 1 \cdot 10^{-4}$ bar this corresponds to a mean free path length of $\lambda = 60$ cm. This pressure is generally taken to be the minimum vacuum for mass spectrometers. The emergency shut-down feature for the cathode (responding to excessive pressure) is almost always set for about $5 \cdot 10^{-4}$ mbar. The desire to be able to use quadrupole spectrometers at higher pressures too, without special pressure convertors, led to the development of the XPR sensor at INFICON (XPR standing for extended pressure range). To enable direct measurement in the range of about $2 \cdot 10^{-2}$ mbar, so important for sputter processes, the rod system was reduced from 12 cm to a length of 2 cm. To ensure that the ions can execute the number of transverse oscillations required for sharp mass separation, this number being about 100, the frequency of the current in the XPR sensor had to be raised from about 2 MHz to approximately 6 times that value, namely to 13 MHz. In spite of the reduction in the length of the rod system, ion yield is still reduced due to dispersion processes at such high pressures. Additional electronic correction is required to achieve perfect depiction of the spectrum. The dimensions of the XPR sensor are so small that it can "hide" entirely inside the tubulation of the connection flange (DN 40, CF) and thus occupies no space in the vacuum chamber proper. Fig. 4.1a shows the size comparison for the normal high-performance sensors with and without the Channeltron SEMP, the normal sensor with channelplate SEMP. Fig. 4.1b shows the XPR sensor. The high vacuum required for the sensor is often generated with a TURBOVAC 50 turbomolecular pump and a D 1.6 B rotary vane pump. With its great compression capacity, a further advantage of the turbomolecular pump when handling high molar mass gases is that the sensor and its cathode are ideally protected from contamination from the direction of the forepump.

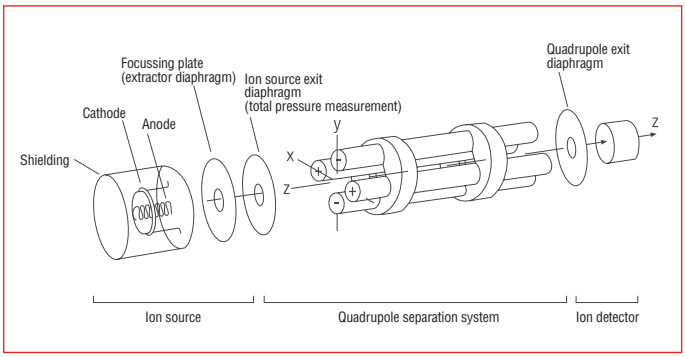


Fig. 4.2 Schematic for quadrupole mass spectrometer

4.3.1 Design of the sensor

One can think of the sensor as having been derived from an extractor measurement system (see Fig. 4.3), whereby the separation system was inserted between the ion source and the ion trap.

4.3.1.1 The normal (open) ion source

The ion source comprises an arrangement of the cathode, anode and several baffles. The electron emission, kept constant, causes partial ionization of the residual gas, into which the ion source is "immersed" as completely as possible. The vacuum in the vicinity of the sensor will naturally be influenced by baking the walls or the cathode. The ions will be extracted through the baffles along the direction of the separation system. One of the baffles is connected to a separate amplifier and – entirely independent of ion separation – provides continuous total pressure measurement (see Fig. 4.4). The cathodes are made of iridium wire and have a thorium oxide coating to reduce the work associated with electron discharge. (For some time now the thorium oxide has gradually been replaced by yttrium oxide.) These coatings reduce the electron discharge work function so that the desired emission flow will be achieved even at lower cathode temperatures. Available for special applications are tungsten cathodes (insensitive to hydrocarbons but sensitive to oxygen) or rhenium cathodes (insensitive to oxygen and hydrocarbons but evaporate slowly during operation due to the high vapor pressure).

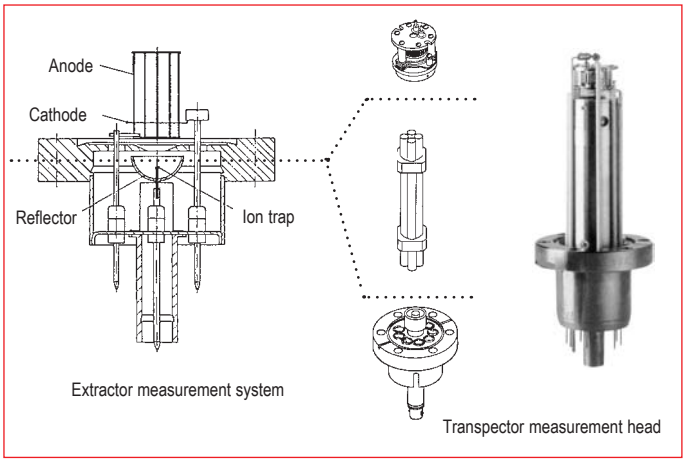


Fig. 4.3 Quadrupole mass spectrometer – Extractor ionization vacuum gauge

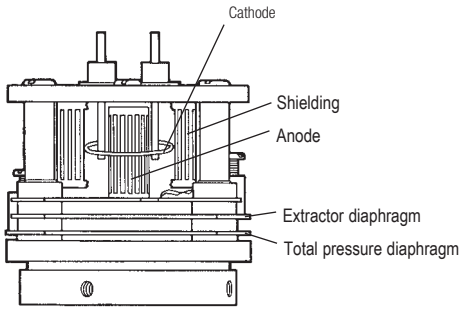


Fig. 4.4 Open ion source

4.3.1.2 The quadrupole separation system

Here the ions are separated on the basis of their mass-to-charge ratio. We know from physics that the deflection of electrically charged particles (ions) from their trajectory is possible only in accordance with their ratio of mass to charge, since the attraction of the particles is proportional to the charge while the inertia (which resists change) is proportional to its mass. The separation system comprises four cylindrical metal rods, set up in parallel and isolated one from the other; the two opposing rods are charged with identical potential. Fig. 4.2 shows schematically the arrangement of the rods and their power supply. The electrical field Φ inside the separation system is generated by superimposing a DC voltage and a high-frequency AC voltage:

$$\Phi = (U + V \lozenge \cos \omega t) \cdot (x^2 - y^2) / r_0^2$$

r_0 = radius of the cylinder which can be inscribed inside the system of rods

Exerting an effect on a single charged ion moving near and parallel to the center line inside the separation system and perpendicular to its movement are the forces

$$F_x = -\frac{2e}{r_0^2} \cdot x \cdot \cos(\omega \cdot t)$$

$$F_y = -\frac{2e}{r_0^2} \cdot y \cdot \cos(\omega \cdot t)$$

$$F_z = 0$$

The mathematical treatment of these equations of motion uses Mathieu's differential equations. It is demonstrated that there are stable and unstable ion paths. With the stable paths, the distance of the ions from the separation system center line always remains less than r_o (passage condition). With unstable paths, the distance from the axis will grow until the ion ultimately collides with a rod surface. The ion will be discharged (neutralized), thus becoming unavailable to the detector (blocking condition).

Even without solving the differential equation, it is possible to arrive at a purely phenomenological explanation which leads to an understanding of the most important characteristics of the quadrupole separation system.

If we imagine that we cut open the separation system and observe the deflection of a singly ionized, positive ion with atomic number M, moving in two planes, which are perpendicular one to the other and each passing through the centers of two opposing rods. We proceed step-by-step and first observe the xz plane (Fig. 4.5, left) and then the yz plane (Fig.4.5, right):

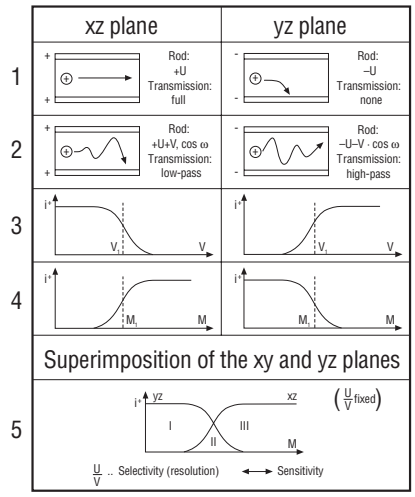


Fig. 4.5 Phenomenological explanation of the separation system

1. Only DC potential U at the rods:

   xz plane (left): Positive potential of +U at the rod, with a repellant effect on the ion, keeping it centered; it reaches the collector (→ passage).

   yz plane (right): Negative potential on the rod -U, meaning that at even the tiniest deviations from the center axis the ion will be drawn toward the nearest rod and neutralized there; it does not reach the collector (→ blocking).

2. Superimposition of high-frequency voltage $V \cdot \cos \omega t$:

   xz plane (left): Rod potential $+U + V \cdot \cos \omega t$. With rising AC voltage amplitude V the ion will be excited to execute transverse oscillations with ever greater amplitudes until it makes contact with a rod and is neutralized. The separation system remains blocked for very large values of V.

   yz plane (right): Rod potential $-U -V \cdot \cos \omega t$. Here again superimposition induces an additional force so that as of a certain value for V the amplitude of the transverse oscillations will be smaller than the clearance between the rods and the ion can pass to the collector at very large V.

3. Ion emission $i^+ = i^+ (V)$ for a fixed mass of M:

   xz plane (left): For voltages of $V < V_1$ the deflection which leads to an escalation of the oscillations is smaller than V_1, i.e. still in the "pass" range. Where $V > V_1$ the deflection will be sufficient to induce escalation and thus blockage.

   yz plane (right): For voltages of $V < V_1$ the deflection which leads to the damping of the oscillations is smaller than V_1, i.e. still in the "block" range. Where $V > V_1$ the damping will be sufficient to settle oscillations, allowing passage.

4. Ion flow $i^+ = i^+ (M)$ for a fixed ratio of U / V:

   Here the relationships are exactly opposite to those for $i^+ = i^+ (V)$ since the influence of V on light masses is greater than on heavy masses.

xz plane: For masses of $M < M_1$ the deflection which results in escalation of the oscillations is greater than at M_1, which means that the ions will be blocked. At $M > M_1$ the deflection is no longer sufficient for escalation, so that the ion can pass.

yz plane: For masses of $M < M_1$ the deflection which results in damping of the oscillations is greater than at M_1, which means that the ion will pass. At $M > M_1$ the damping is not sufficient to calm the system and so the ion is blocked.

5. Combination of the xz and yz planes. In the superimposition of the ion currents $i^+ = i^+ (M)$ for both pairs of rods (U / V being fixed) there are three important ranges:

   Range I: No passage for M due to the blocking behavior of the xz pair of rods.

   Range II: The pass factor of the rod systems for mass M is determined by the U/V ratio (other ions will not pass). We see that great permeability (corre- sponding to high sensitivity) is bought at the price of low selectivity (= resolution, see Section 4.5). Ideal adjustment of the separation system thus requires a compromise between these two properties. To achieve constant resolution, the U/V ratio will remain constant over the entire measurement range. The "atomic number" M (see 4.6.1) of the ions which can pass through the separation system must satisfy this condition:

$$\frac{m}{e} \approx M = \frac{V}{14.438 \cdot f^2 \cdot r_0^2}$$

   V = High-frequency amplitude,
   r_0 = Quadrupole inscribed radius
   f = High-frequency

   As a result of this linear dependency there results a mass spectrum with linear mass scale due to simultaneous, proportional modification of U and V.

   Range III: M cannot pass, due to the blocking characteristics of the yz pair of rods.

4.3.1.3 The measurement system (detector)

Once they have left the separation system the ions will meet the ion trap or detector which, in the simplest instance, will be in the form of a Faraday cage (Faraday cup). In any case the ions which impinge on the detector will be neutralized by electrons from the ion trap. Shown, after electrical amplification, as the measurement signal itself is the corresponding "ion emission stream". To achieve greater sensitivity, a secondary electron multiplier pickup (SEMP) can be employed in place of the Faraday cup.

Channeltrons or Channelplates can be used as SEMPs. SEMPs are virtually inertia-free amplifiers with gain of about 10^{+6} at the outset; this will indeed drop off during the initial use phase but will then become virtually constant over a long period of time. Fig. 4.6 shows at the left the basic configuration of a Faraday ion trap and, on the right, a section through a Channeltron. When recording spectra the scanning period per mass line t_0 and the time constants of the amplifier t should satisfy the condition that $t_0 = 10\ \tau$. In modern devices such as the TRANSPECTOR the otherwise unlimited selection of the scanning period and the amplifier time constants will be restricted by microprocessor control to logical pairs of values.

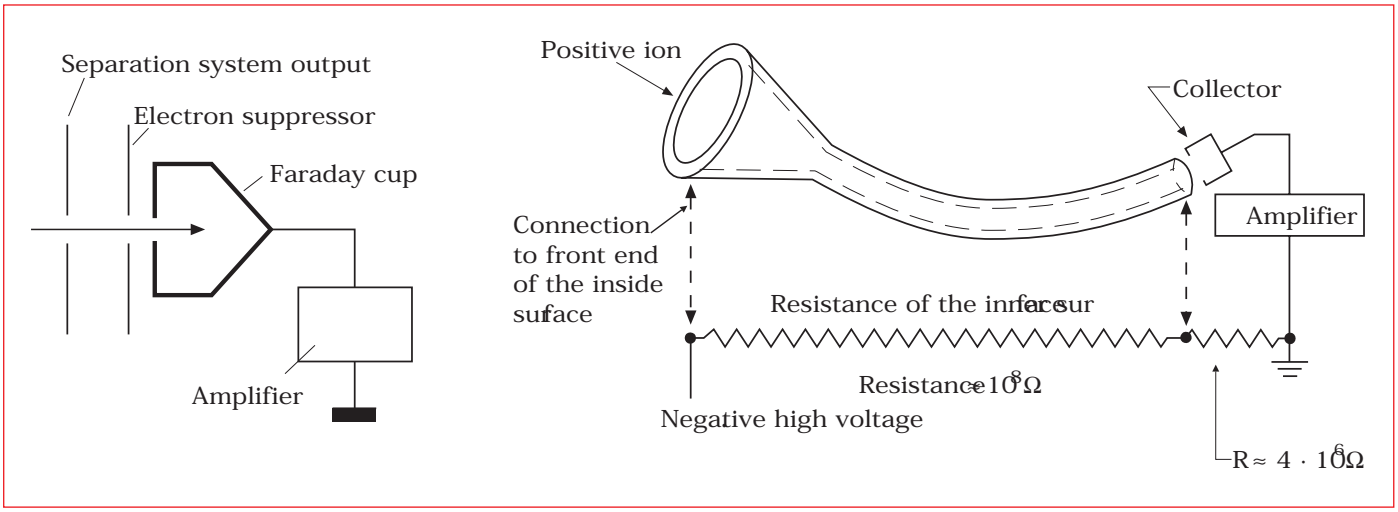


Fig. 4.6 Left: Principle of the Faraday cup; Right: Configuration of the Channeltron

4.4 Gas admission and pressure adaptation

4.4.1 Metering valve

The simplest way to adapt a classical mass spectrometer to pressures exceeding $1 \cdot 10^{-4}$ mbar is by way of a metering valve. The inherent disadvantage is, however, that since the flow properties are not unequivocally defined, a deviation from the original gas composition might result.

4.4.2 Pressure converter

In order to examine a gas mix at total pressure exceeding $1 \cdot 10^{-4}$ mbar it is necessary to use pressure converters which will not segregate the gases. Figure 4.7 is used to help explain how such a pressure converter works:

a. Process pressure < 1 mbar: Single-stage pressure converter. Gas is allowed to pass out of the vacuum vessel in molecular flow, through a diaphragm with conduc- tance value L_2 and into the "sensor chamber" (with its own high vacuum system). Molecular flow causes segregation but this will be independent of the pressure level (see Section 1.5). A second diaphragm with molecular flow, located between the sensor chamber and the turbomolecular pump, will compensate for the segregation occurring at L_2.

b. Process pressure > 1 mbar: Two-stage pressure converter. Using a small (rotary vane) pump a laminar stream of gas is diverted from the rough vacuum area through a capillary or diaphragm (conductance value L_3). Prior to entry into the pump, at a pressure of about 1 mbar, a small part of this flow is again allowed to enter the sensor chamber through the diaphragm with conductance value L_2, again as molecular flow.

A falsification of the gas composition resulting from adsorption and condensation can be avoided by heating the pressure converter and the capillary.

To evaluate the influence on the gas composition by the measurement unit itself, information on the heating temperature, the materials and surface areas for the metallic, glass and ceramic components will be needed along with specifications on the material and dimensions of the cathode (and ultimately regarding the electron impact energy for the ion source as well).

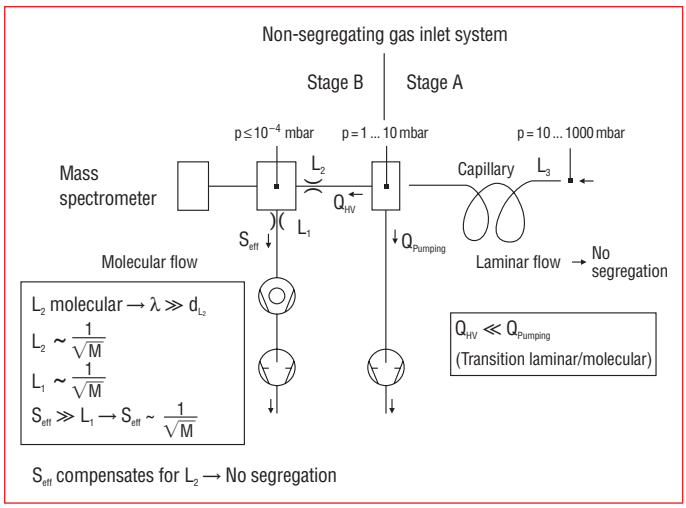


Fig. 4.7 Principle of the pressure converter (stage B only in the single-stage version and stages A and B in two-stage units)

4.4.3 Closed ion source (CIS)

In order to curb – or avoid entirely – influences which could stem from the sensor chamber or the cathode (e.g. disturbance of the $CO\text{-}CO_2$ equilibrium by heating the cathode) a closed ion source (CIS) will be used in many cases.

The CIS is divided into two sections: a cathode chamber where the electrons are emitted, and an impact chamber, where the impact ionization of the gas particles takes place. The two chambers are pumped differentially: the pressure in the cathode chamber comes to about 10^{-5} mbar, that in the impact room about 10^{-3} mbar. The gas from the vacuum chamber is allowed to pass into the impact chamber by way of a metal-sealed, bakeable valve (pressure converter, ultrahigh vacuum technology). There high-yield ionization takes place at about 10^{-3} mbar. The electrons exerting the impact are emitted in the cathode chamber at about 10^{-5} mbar and pass through small openings from there into the impact chamber. The signal-to-noise ratio (residual gas) via à vis the open ion source will be increased overall by a factor of 10^{+3} or more. Figure 4.8 shows the fundamental difference between the configurations for open and closed ion sources for a typical application in sputter technology. With the modified design of the CIS compared with the open ion source in regard to both the geometry and the electron energy (open ion source 102 eV, CIS 75 or 35 eV), different fragment distribution patterns may be found where a lower electron energy level is selected. For example, the argon36++ isotope at mass of 18 cannot be detected at electron energy of less than 43.5 eV and can therefore not falsify the detection of H_2O+ at mass 18 in the sputter processes using argon as the working gas – processes which are of great importance in industry.

4.4.4 Aggressive gas monitor (AGM)

In many cases the process gas to be examined is so aggressive that the cathode would survive for only a short period of time. The AGM uses the property of laminar flow by way of which there is no "reverse" flow of any kind. Controlled with a separate AGM valve, a part of the working gas fed to the processes is introduced as "purging gas", ahead of the pressure converter, to the TRANSPECTOR; this sets up a flow toward the vacuum chamber. Thus process gas can reach the TRANSPECTOR only with the AGM valve closed. When the valve is open the TRANSPECTOR sees only pure working gas. Fig. 4.9 shows the AGM principle.

Mass spectrometry

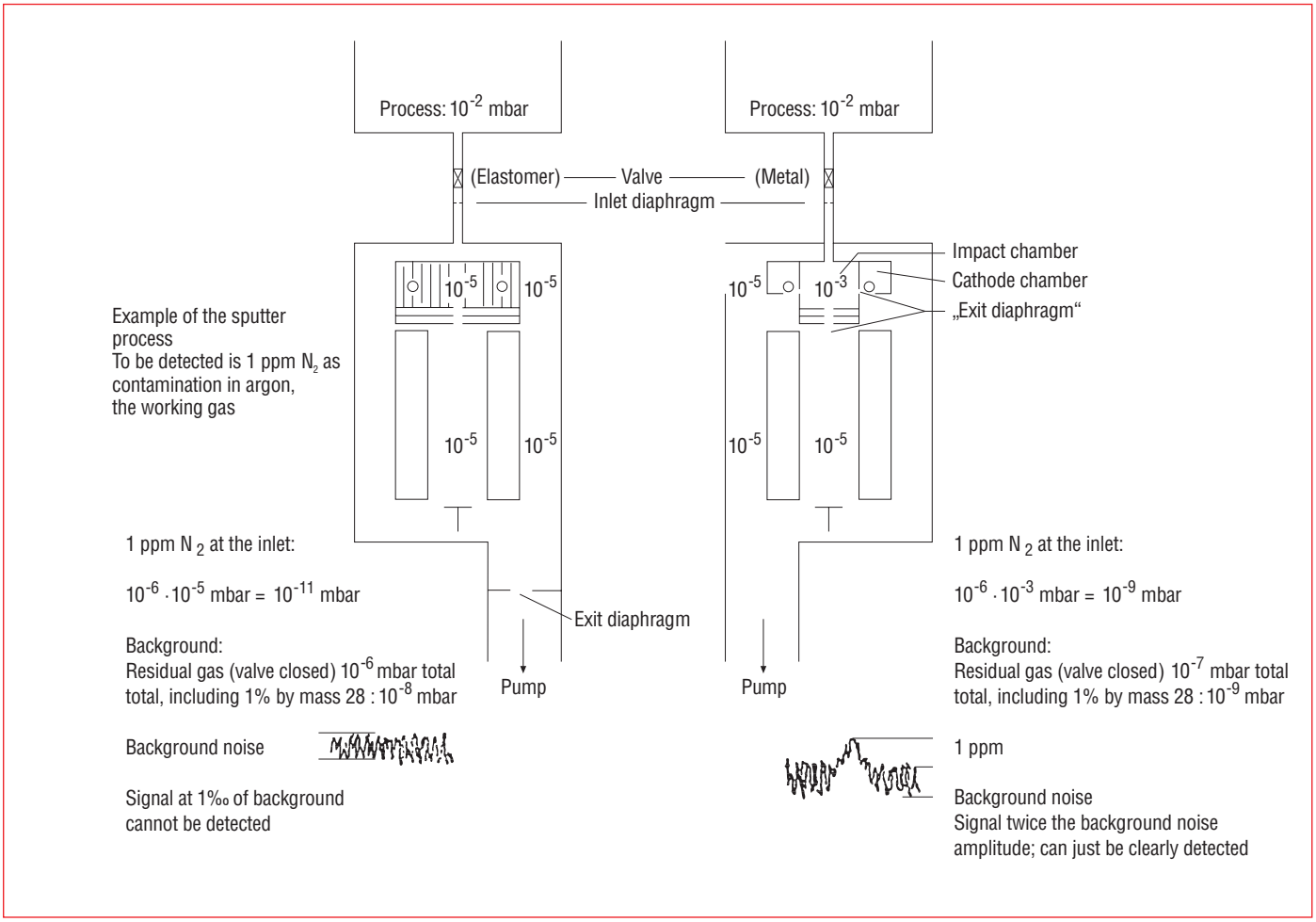


Fig. 4.8 Open ion source (left) and closed ion source (right)

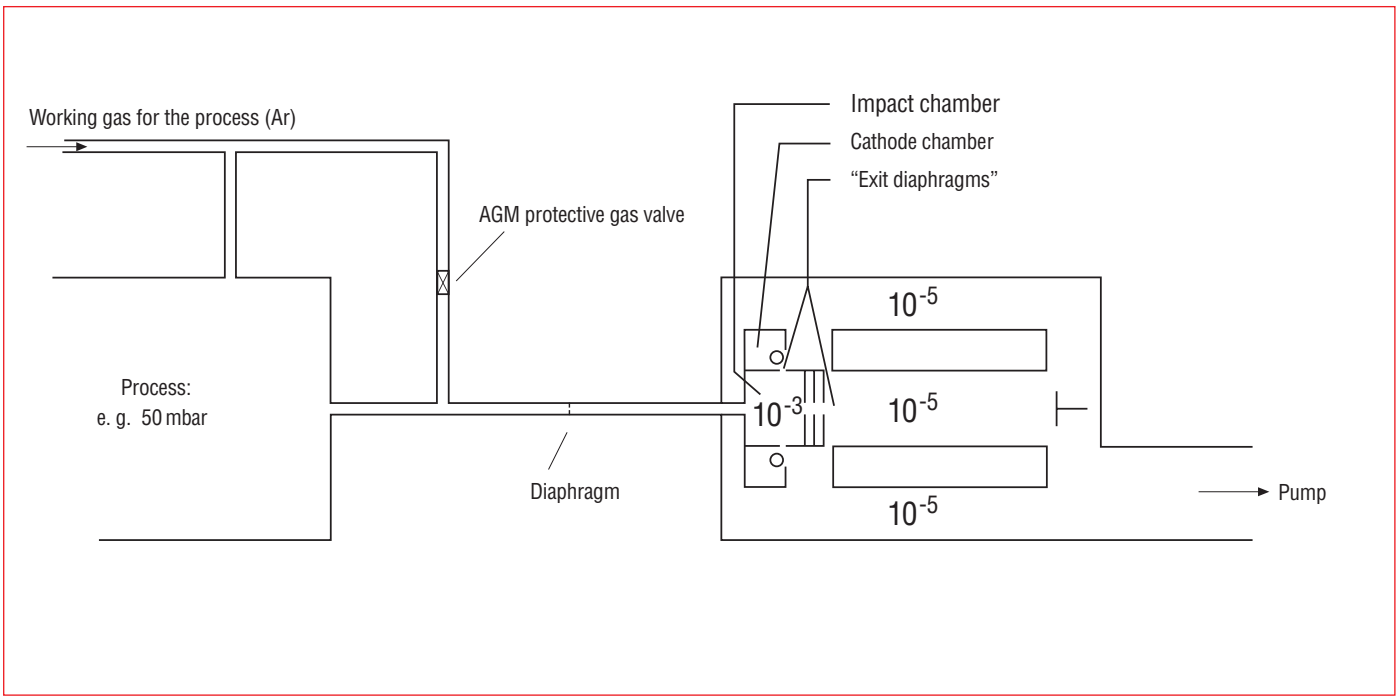


Fig. 4.9 Principle behind the aggressive gas monitor (AGM)

4.5 Descriptive values in mass spectrometry (specifications)

A partial pressure measurement unit is characterized essentially by the following properties (DIN 28 410):

4.5.1 Line width (resolution)

The line width is a measure of the degree to which differentiation can be made between two adjacent lines of the same height. The resolution is normally indicated. It is defined as: R = M / ΔM and is constant for the quadrupole spectrometer across the entire mass range, slightly greater than 1 or ΔM < 1.

Often an expression such as “unit resolution with 15% valley” is used. This means that the “bottom of the valley” between two adjacent peaks of identical height comes to 15 % of the height of the peak or, put another way, at 7.5 % of its peak height the line width DM measured across an individual peak equals 1 amu (atomic mass unit); see in this context the schematic drawing in Fig. 4.10.

4.5.2 Mass range

The mass range is characterized by the atomic numbers for the lightest and heaviest ions with a single charge which are detected with the unit.

4.5.3 Sensitivity

Sensitivity E is the quotient of the measured ion flow and the associated partial pressure; it is normally specified for argon or nitrogen:

$$E = \frac{i^+}{p_G}\left(\frac{A}{mbar}\right) \qquad (4.1)$$

Typical values are:

Faraday cup: $E = 1 \cdot 10^{-4}\,\frac{A}{mbar}$

SEMP: $E = 1 \cdot 10^{+2}\,\frac{A}{mbar}$

4.5.4 Smallest detectable partial pressure

The smallest detectable partial pressure is defined as a ratio of noise amplitude to sensitivity:

$$P_{min} = \frac{\Delta \cdot i_R^+}{E}\,(mbar)$$

$\Delta \cdot i_R^+$ = Noise amplitude

Example (from Fig. 4.11):

Sensitivity $E = 1 \cdot 10^{-4}\,\frac{A}{mbar}$

Noise amplitude $\Delta \cdot i_R^+ = 4 \cdot 10^{-14}$ A

$$p_{min}(FC) = \frac{4 \cdot 10^{-14} A}{1 \cdot 10^{-4} A/mbar} = 4 \cdot 10^{-10}\ mbar$$

4.5.5 Smallest detectable partial pressure ratio (concentration)

The definition is:

SDPPR = p_{min} / p_{Σ} (ppm)

This definition, which is somewhat “clumsy” for practical use, is to be explained using the detection of argon36 in the air as the example: Air contains 0.93 % argon by volume; the relative isotope frequency of Ar_{40} to Ar_{36} is 99.6 % to 0.337 %. Thus the share of Ar_{36} in the air can be calculated as follows:

$0.93 \cdot 10^{-2} \cdot 0.337 \cdot 10^{-2} = 3.13 \cdot 10^{-5} = 31.3$ ppm

Figure 4.11 shows the screen print-out for the measurement. The peak height for Ar_{36} in the illustration is determined to be $1.5 \cdot 10^{-13}$ A and noise amplitude $\Delta \cdot i_R^+$ to be $4 \cdot 10^{-14}$ A. The minimum detectable concentration is that concentration at which the height of the peak is equal to the noise amplitude. This results in the smallest measurable peak height being $1.5 \cdot 10^{-13}$ A/$2.4 \cdot 10^{-14}$ A = 1.875. The smallest detectable concentration is then derived from this by calculation to arrive at:

$31.3 \cdot 10^{-6} / 1.875 = 16.69 \cdot 10^{-6} = 16.69$ ppm.

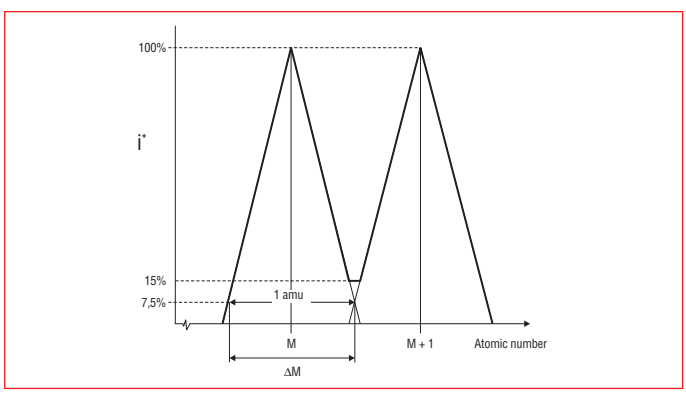


Fig. 4.10 Line width – 15 % valley

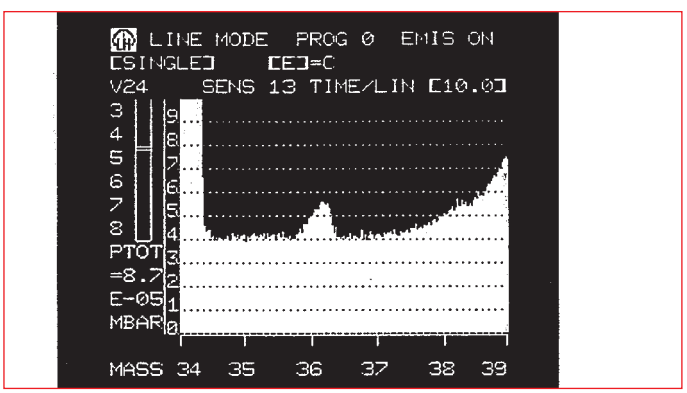


Fig. 4.11 Detection of $Argon_{36}$

4.5.6 Linearity range

The linearity range is that pressure range for the reference gas (N_2, Ar) in which sensitivity remains constant within limits which are to be specified (± 10 % for partial pressure measurement devices).

In the range below $1 \cdot 10^{-6}$ mbar the relationship between the ion flow and partial pressure is strictly linear. Between $1 \cdot 10^{-6}$ mbar and $1 \cdot 10^{-4}$ mbar there are minor deviations from linear characteristics. Above $1 \cdot 10^{-4}$ mbar these deviations grow until, ultimately, in a range above 10^{-2} mbar the ions for the dense gas atmosphere will no longer be able to reach the ion trap. The emergency shut-down for the cathode (at excessive pressure) is almost always set for $5 \cdot 10^{-4}$ mbar. Depending on the information required, there will be differing upper limits for use.

In analytical applications, $1 \cdot 10^{-6}$ mbar should not be exceeded if at all possible. The range from $1 \cdot 10^{-6}$ mbar to $1 \cdot 10^{-4}$ mbar is still suitable for clear depictions of the gas composition and partial pressure regulation (see Fig. 4.12).

4.5.7 Information on surfaces and amenability to bake-out

Additional information required to evaluate a sensor includes specifications on the bake-out temperature (during measurement or with the cathode or SEMP switched off), materials used and surface areas of the metal, glass and ceramic components and the material and dimensions for the cathode; data is also needed on the electron impact energy at the ion source (and on whether it is adjustable). These values are critical to uninterrupted operation and to any influence on the gas composition by the sensor itself.

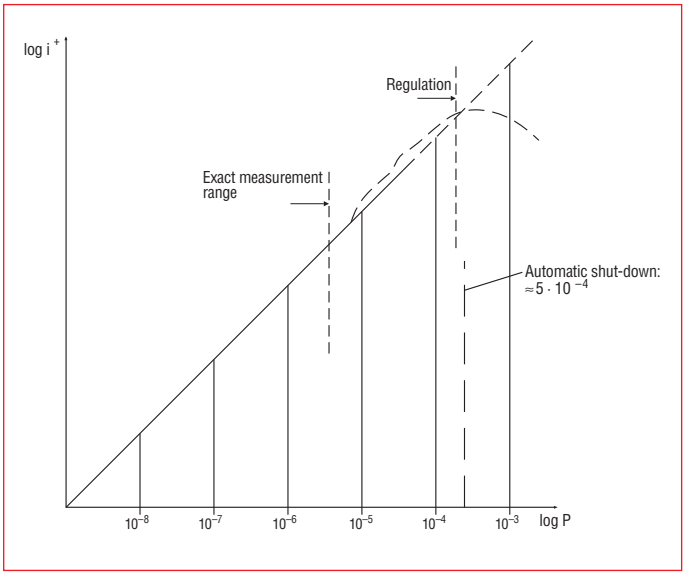


Fig. 4.12 Qualitative linearity curve

4.6 Evaluating spectra

4.6.1 Ionization and fundamental problems in gas analysis

Continuous change in the voltages applied to the electrodes in the separation syssem ("scanning") gives rise to a relationship between the ion flow I+ and the "atomic number" which is proportional to the m/e ratio and expressed as:

$$M = \frac{M_r}{n_e} \qquad (4.2)$$

(M_r = relative molar mass, n_e = number of elementary charges e)

This is the so-called mass spectrum, $i^+ = i^+(M)$. The spectrum thus shows the peaks i+ as ordinates, plotted against the atomic number M along the abscissa. One of the difficulties in interpreting a mass spectrum such as this is due to the fact that one and the same mass as per the equation (4.2) may be associated with various ions. Typical examples, among many others, are: The atomic number M = 16 corresponds to CH_4^+ and O_2^{++}; M = 28 for CO^+, N_2^+ and C_2H^+! Particular attention must therefore be paid to the following points when evaluating spectra:

1) In the case of **isotopes** we are dealing with differing positron counts in the nucleus (mass) of the ion at identical nuclear charge numbers (gas type). Some values for relative isotope frequency are compiled in Table 4.2.

Element	Ordinal-number	Atomic number	Relative frequency
H	1	1 2	99.985 0.015
He	2	3 4	0.00013 ≈ 100.0
B	5	10 11	19.78 80.22
C	6	12 13	98.892 1.108
N	7	14 15	99.63 0.37
O	8	16 17 18	99.759 0.0374 0.2039
F	9	19	100.0
Ne	10	20 21 22	90.92 0.257 8.82
Na	11	23	100.0

Table 4.2 Relative frequency of isotopes

Element	Ordinal-number	Atomic number	Relative frequency
Al	13	27	100.0
Si	14	28	92.27
		29	4.68
		30	3.05
P	15	31	100.0
S	16	32	95.06
		33	0.74
		34	4.18
		36	0.016
Cl	17	35	75.4
		37	24.6
Ar	18	36	0.337
		38	0.063
		40	99.60
Kr	36	78	0.354
		80	2.27
		82	11.56
		83	11.55
		84	56.90
		86	17.37
Xe	54	124	0.096
		126	0.090
		128	1.919
		129	26.44
		130	4.08
		131	21.18
		132	26.89
		134	10.44
		136	8.87

Table 4.2 Relative frequency of isotopes

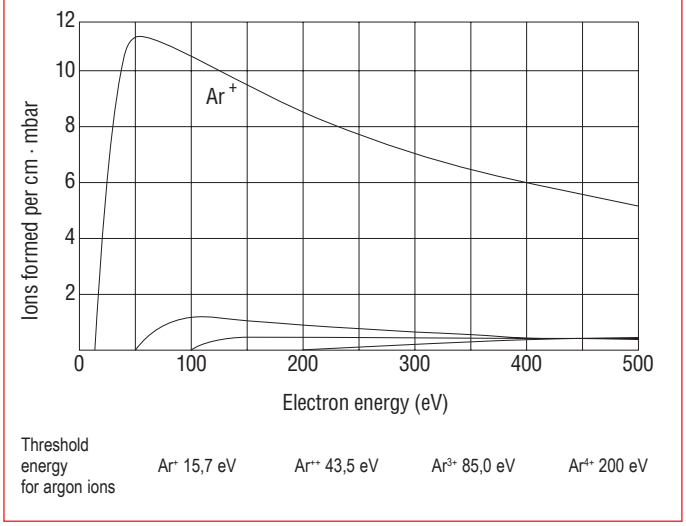


Fig. 4.13 Number of the various Ar ions produced, as a factor of electron energy level

2) Depending on the energy of the impacting electrons (equalling the potential differential, cathode – anode), ions may be either singly or multiply ionized. For example, one finds Ar+ at mass of 40, Ar^{++} at mass of 20 and Ar^{+++} at mass of 13.3. At mass of 20 one will, however, also find neon, Ne^{+}. There are threshold energy levels for the impacting electrons for all ionization states for every type of gas, i.e., each type of ion can be formed only above the associated energy threshold. This is shown for Ar in Fig. 4.13.

3) **Specific ionization** of the various gases S_{gas}, this being the number of ions formed, per cm and mbar, by collisions with electrons; this will vary from one type of gas to the next. For most gases the ion yield is greatest at an electron energy level between about 80 and 110 eV; see Fig. 4.14.

In practice the differing ionization rates for the individual gases will be taken into account by standardization against nitrogen; **relative ionization probabilities (RIP)** in relationship to nitrogen will be indicated (Table 4.3).

4) Finally, gas molecules are often broken down into fragments by ionization. The **fragment distribution patterns** thus created are the so-called characteristic spectra (**fingerprint, cracking pattern**). **Important:** In the tables the individual fragments specified are standardized either against the maximum peak (in % or ‰ of the highest peak) or against the total of all peaks (see the examples in Table 4.4).

Both the nature of the fragments created and the possibility for multiple ionization will depend on the geometry (differing ion number, depending on the length of the ionization path) and on the energy of the impacting electrons (threshold energy for certain types of ions). Table values are always referenced to a certain ion source with a certain electron energy level. This is why it is difficult to compare the results obtained using devices made by different manufacturers.

Often the probable partial pressure for one of the masses involved will be estimated through critical analysis of the spectrum. Thus the presence of air in the vacuum vessel (which may indicate a leak) is manifested by the detection of a quantity of O_2^+ (with mass of 32) which is about one-quarter of the share of N_2^+ with its mass of 28. If, on the other hand, no oxygen is

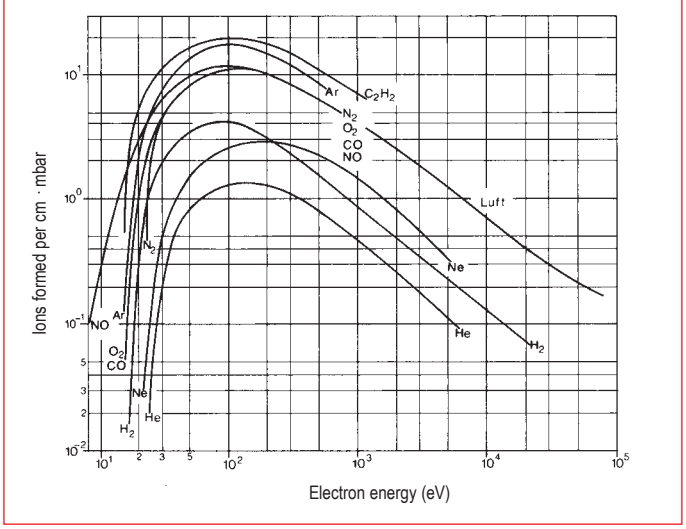


Fig. 4.14 Specific ionization S for various gases by electrons exhibiting energy level E

Type of gas	Symbol	RIP	Type of gas	Symbol	RIP
Acetone (Propanone)	$(CH_3)_2CO$	3.6	Hydrogen chloride	HCl	1.6
Air		1.0	Hydrogen fluoride	HF	1.4
Ammonia	NH_3	1.3	Hydrogen iodide	HI	3.1
Argon	Ar	1.2	Hydrogen sulfide	H_2S	2.2
Benzene	C_6H_6	5.9	Iodine	I_2	
Benzoic acid	C_6H_5COOH	5.5	Krypton	Kr	1.7
Bromine	Br	3.8	Lithium	Li	1.9
Butane	C_4H_{10}	4.9	Methane	CH_4	1.6
Carbon dioxide	CO_2	1.4	Methanol	CH_3OH	1.8
Carbon disulfide	CS_2	4.8	Neon	Ne	0.23
Carbon monoxide	CO	1.05	Nitrogen	N_2	1.0
Carbon tetrachloride	CCl_4	6.0	Nitrogen oxide	NO	1.2
Chlorobenzene	C_6H_4Cl	7.0	Nitrogen dioxide	N_2O	1.7
Chloroethane	C_2H_3Cl	4.0	Oxygen	O_2	1.0
Chloroform	$CHCl_3$	4.8	n-pentane	C_5H_{17}	6.0
Chlormethane	CH_3Cl	3.1	Phenol	C_6H_5OH	6.2
Cyclohexene	C_6H_{12}	6.4	Phosphine	PH_3	2.6
Deuterium	D_2	0.35	Propane	C_3H_8	3.7
Dichlorodifluoromethane	CCl_2F_2	2.7	Silver perchlorate	$AgClO_4$	3.6
Dichloromethane	CH_2Cl_2	7.8	Tin iodide	SnI_4	6.7
Dinitrobenzene	$C_6H_4(NO_2)_2$	7.8	Sulfur dioxide	SO_2	2.1
Ethane	C_2H_6	2.6	Sulfur hexafluoride	SF_6	2.3
Ethanol	C_2H_5OH	3.6	Toluene	$C_6H_5CH_3$	6.8
Ethylene oxide	$(CH_2)_2O$	2.5	Trinitrobenzene	$C_6H_3(NO_2)_3$	9.0
Helium	He	0.14	Water vapor	H_2O	11.0
Hexane	C_6H_{14}	6.6	Xenon	Xe	3.0
Hydrogen	H_2	0.44	Xylols	$C_6H_4(CH_3)_2$	7.8

Table 4.3 Relative ionization probabilities (RIP) vis à vis nitrogen, electron energy 102 eV

Electron energy :			75 eV (PGA 100)		102 eV (Transpector)	
Gas	Symbol	Mass	Σ = 100 %	Greatest peak = 100 %	Σ = 100 %	Greatest peak = 100 %
Argon	Ar	40	74.9	100	90.9	100
		20	24.7	33.1	9.1	10
		36				0.3
Carbon dioxide	CO_2	45	0.95	1.3	0.8	1
		44	72.7	100	84	100
		28	8.3	11.5	9.2	11
		16	11.7	16.1	7.6	9
		12	6.15	8.4	5	6
Carbon monoxide	CO	29	1.89	2.0	0.9	1
		28	91.3	100	92.6	100
		16	1.1	1.2	1.9	2
		14	1.7	1.9		0.8
		12	3.5	3.8	4.6	5
Neon	Ne	22	9.2	10.2	0.9	11
		20	89.6	100	90.1	100
		10	0.84	0.93	9	4
Oxygen	O_2	34	0.45	0.53		
		32	84.2	100	90.1	100
		16	15.0	17.8	9.9	11
Nitrogen	N2	29	0.7	0.8	0.9	1
		28	86.3	100	92.6	100
		14	12.8	15	6.5	12
Water vapor	H_2O	19	1.4	2.3		
		18	60	100	74.1	100
		17	16.1	27	18.5	25
		16	1.9	3.2	1.5	2
		2	5.0	8.4	1.5	2
		1	15.5	20	4.4	6

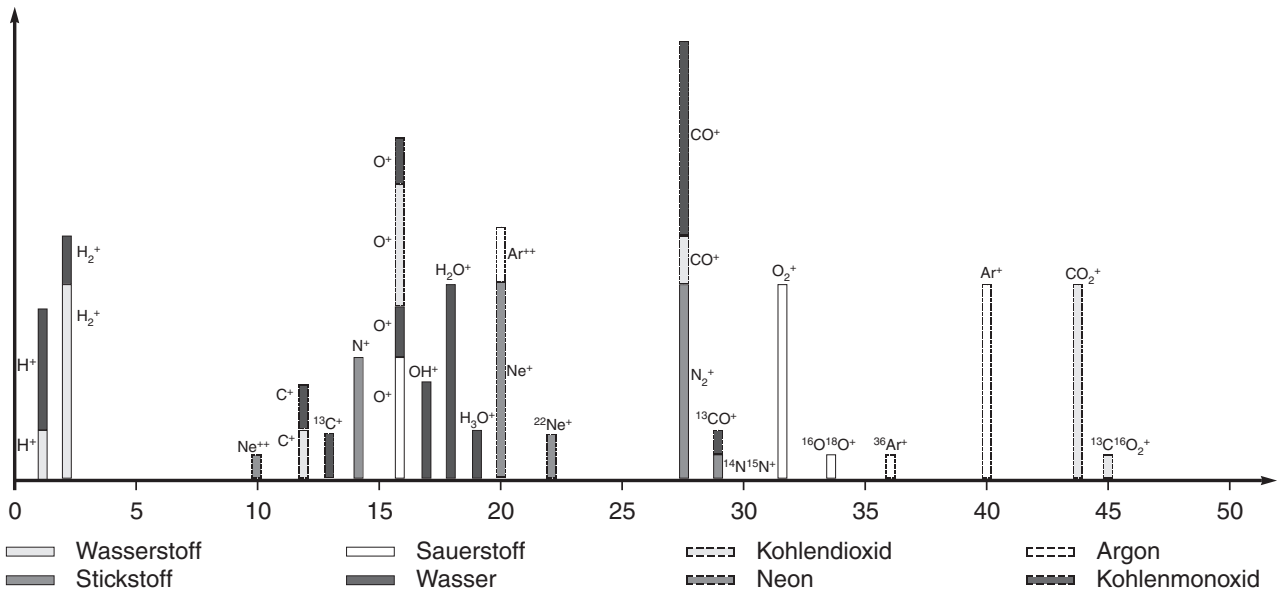
Table 4.4 Fragment distribution for certain gases at 75 eV and 102 eV

No	Gas	Symbol	1 = 100	2	3	4	5	6
1	Acetone	$(CH_3)_2CO$	43/100	15/42	58/20	14/10	27/19	42/8
2	Air		28/100	32/27	14/6	16/3	40/1	-
3	Ammonia	NH_3	17/100	16/80	15/8	14/2	-	-
4	Argon	Ar	40/100	20/10	-	-	-	-
5	Benzene	C_6H_6	78/100	77/22	51/18	50/17	52/15	39/10
6	Carbon dioxide	CO_2	44/100	28/11	16/9	12/6	45/1	22/1
7	Carbon monoxide	CO	28/100	12/5	16/2	29/1	-	-
8	Carbon tetrachloride	CCl_4	117/100	119/91	47/51	82/42	35/39	121/29
9	Carbon tetrafluoride	CF_4	69/100	50/12	31/5	19/4	-	-
10	Diff. pump oil, DC 705		78/100	76/83	39/73	43/59	91/32	-
11	Diff. pump oil, Fomblin		69/100	20/28	16/16	31/9	97/8	47/8
12	Diff. pump oil, PPE		50/100	77/89	63/29	62/27	64/21	38/7
13	Ethanol	CH_3CH_2OH	31/100	45/34	27/24	29/23	46/17	26/8
14	Halocarbon 11	CCl_3F	101/100	103/60	35/16	66/15	47/12	31/10
15	Halocarbon 12	CCl_2F_2	85/100	87/32	50/16	35/12	-	-
16	Halocarbon 13	$CClF_3$	69/100	85/15	50/14	31/9	35/7	87/5
17	Halocarbon 14	CF_4	69/100	12/7	19/6	31/5	50/8	-
18	Halocarbon 23	CHF_3	51/100	31/58	69/40	50/19	52/1	21/1
19	Halocarbon 113	$C_2C_{13}F_3$	101/100	103/62	85/55	31/50	151/41	153/25
20	Helium	He	4/100	-	-	-	-	-
21	Heptane	C_7H_{16}	43/100	41/62	29/49	27/40	57/34	71/28
22	Hexane	C_6H_{14}	41/100	43/92	57/85	29/84	27/65	56/50
23	Hydrogen	H_2	2/100	1/5	-	-	-	-
24	Hydrogen sulfide	H_2S	34/100	32/44	33/42	36/4	35/2	-
25	Isopropyl alcohol	C_3H_8O	45/100	43/16	27/16	29/10	41/7	39/6
26	Krypton	Kr	84/100	86/31	83/20	82/20	80/4	-
27	Methane	CH_4	16/100	15/85	14/16	13/8	1/4	12/2
28	Mehtyl alcohol	CH_3OH	31/100	29/74	32/67	15/50	28/16	2/16
29	Methyl ethyl ketone	C_4H_8O	43/100	29/25	72/16	27/16	57/6	42/5
30	Mechanical pump oil		43/100	41/91	57/73	55/64	71/20	39/19
31	Neon	Ne	20/100	22/10	10/1	-	-	-
32	Nitrogen	N_2	28/100	14/7	29/1	-	-	-
33	Oxygen	O_2	32/100	16/11	-	-	-	-
34	Perfluorokerosene		69/100	119/17	51/12	131/11	100/5	31/4
35	Perfluor-tributylamine	$C_{12}F_{27}N$	69/100	131/18	31/6	51/5	50/3	114/2
36	Silane	SiH_4	30/100	31/80	29/31	28/28	32/8	33/2
37	Silicon tetrafluoride	SiF_4	85/100	87/12	28/12	33/10	86/5	47/5
38	Toluene	$C_6H_5CH_3$	91/100	92/62	39/12	65/6	45.5/4	51/4
39	Trichloroethane	$C_2H_3Cl_3$	97/100	61/87	99/61	26/43	27/31	63/27
40	Trichloroethylene	C_2HCl_3	95/100	130/90	132/85	97/64	60/57	35/31
41	Trifluoromethane	CHF_3	69/100	51/91	31/49	50/42	12/4	-
42	Turbomolecular pump oil		43/100	57/88	41/76	55/73	71/52	69/49
43	Water vapor	H_2O	18/100	17/25	1/6	16/2	2/2	-
44	Xenon	Xe	132/100	129/98	131/79	134/39	136/33	130/15

Table 4.5 Spectrum library of the 6 highest peaks for the TRANSPECTOR

detected in the spectrum, then the peak at atomic number 28 would indicate carbon monoxide. In so far as the peak at atomic number 28 reflects the CO^+ fragment of CO_2 (atomic number 44), this share is 11 % of the value measured for atomic number 44 (Table 4.5). On the other hand, in all cases where nitrogen is present, atomic number 14 (N_2^{++}) will always be found in the spectrum in addition to the atomic number 28 (N_2^+); in the case of carbon monoxide, on the other hand, there will always appear – in addition to CO^+ – the fragmentary masses of 12 (C^+) and 16 (O_2^{++})).

Figure 4.15 uses a simplified example of a “model spectrum” with superimpositions of hydrogen, nitrogen, oxygen, water vapor, carbon monoxide, carbon dioxide, neon and argon to demonstrate the difficulties involved in evaluating spectra.



Evaluation problems: The peak at atomic number 16 may, for example, be due to oxygen fragments resulting from O_2, H_2O, CO_2 and CO; the peak at atomic number 28 from contributions by N_2 as well as by CO and CO as a fragment of CO_2; the peak at atomic number 20 could result from singly ionized Ne and double-ionized Ar.

Fig. 4.15 Model spectrum

4.6.2 Partial pressure measurement

The number of ions i^+_{gas} produced from a gas in the ion source is proportional to the emission current i^-, to the specific ionization S_{gas}, to a geometry factor f representing the ionization path inside the ionization source, to the relative ionization probability RIP_{gas}, and to the partial pressure p_{gas}. This number of ions produced is, by definition, made equal to the sensitivity Egas times the partial pressure p_{gas}:

$$i^+_{gas}\,(\text{produced}) = i^- \cdot S_{gas} \cdot f \cdot RIP_{gas} \cdot p_{gas}$$

$$= E_{gas} \cdot p_{gas}$$

due to $RIP_{N2} = 1$

EN_2 is equal to $i^- \cdot SN_2 \cdot f$

and E_{gas} is equal to $EN_2 \cdot RIP_{gas}$

Almost all gases form fragments during ionization. To achieve quantitative evaluation one must either add the ion flows at the appropriate peaks or measure (with a known fragment factor [FF]) one peak and calculate the overall ion flow on that basis:

$$i^+_{gas}(\text{produced}) = i^+_{gas,m_1} + i^+_{gas,m_2} + \ldots = \frac{i^+_{gas,m_1}}{BF_{gas,m_1}}$$

$$= \frac{i^+_{gas,m_2}}{BF_{gas,m_2}} = \ldots = E_{gas} \cdot p_{gas}$$

In order to maintain the number of ions arriving at the ion trap, it is necessary to multiply the number above with the transmission factor TF(m), which will be dependent on mass, in order to take into account the permeability of the separation system for atomic number m (analogous to this, there is the detection factor for the SEMP; it, however, is often already contained in TF). The transmission factor (also: ion-optical transmission) is thus the quotient of the ions measured and the ions produced.

Thus

$$p_{gas} = \frac{i^+_{gas,m_2}(\text{produced})}{BF_{gas,m_2} \cdot E_{gas}}$$

$$\Rightarrow p_{gas} = \frac{i^+_{gas,m_2}(\text{measured})}{BF_{gas,m_2} \cdot E_{gas} \cdot TF(m)}$$

and with

a $E_{gas} = E_{N2} \cdot RIP_{gas}$

the ultimate result is:

$$p_{gas} = i^+_{gas,m_2}(\text{measured}) \cdot \frac{1}{E_{N_2}} \cdot \frac{1}{BF_{gas,m_2} \cdot RIP_{gas} \cdot TF(m)} \qquad (4.3)$$

The partial pressure is calculated from the ion flow measured for a certain fragment by multiplication with two factors. The first factor will depend only on the nitrogen sensitivity of the detector and thus is a constant for the device. The second will depend only on the specific ion properties.

These factors will have to be entered separately for units with direct partial pressure indication (at least for less common types of ions).

4.6.3 Qualitative gas analysis

The analysis of spectra assumes certain working hypotheses:

1. Every type of molecule produces a certain, constant mass spectrum or fragment spectrum which is characteristic for this type of molecule (fingerprint, cracking pattern).
2. The spectrum of every mixture of gases is the same as would be found

through linear superimposition of the spectra of the individual gases. The height of the peaks will depend on the gas pressure.

3. The ion flow for each peak is proportional to the partial pressure of each component responsible for the peak. Since the ion flow is proportional to the partial pressure, the constant of proportionality (sensitivity) varies from one gas to the next.

Although these assumptions are not always correct (see Robertson: Mass Spectrometry) they do represent a useful working hypothesis.

In qualitative analysis, the unknown spectrum is compared with a known spectrum in a library. Each gas is “definitively determined” by its spectrum. The comparison with library data is a simple pattern recognition process. Depending on the availability, the comparison may be made using any of a number ancillary aids. So, for example, in accordance with the position, size and sequence of the five or ten highest peaks. Naturally, comparison is possible only after the spectrum has been standardized, by setting the height of the highest line equal to 100 or 1000 (see Table 4.5 as an example).

The comparison can be made manually on the basis of collections of tables (for example, A. Cornu & R. Massot: Compilation of Mass Spectral Data) or may be effected with computer assistance; large databases can be used (e.g. Mass Spectral Data Base, Royal Society of Chemistry, Cambridge).

When making comparisons with library information, it is necessary to pay attention to whether identical ion sources or at least identical electron impact energies were used.

All these capabilities are, however, generally too elaborate for the problems encountered in vacuum technology. Many commercial mass spectrometers can show a number of library spectra in the screen so that the user can see immediately whether the “library substance” might be contained in the substance measured. Usually the measured spectrum was the result of a mix of gases and it is particularly convenient if the screen offers the capacity for subtracting (by way of trial) the spectra of individual (or several) gases from the measured spectrum. The gas can be present only when the subtraction does not yield any negative values for the major peaks. Figure 4.16 shows such a step-by-step subtraction procedure using the Transpector-Ware software.

Regardless of how the qualitative analysis is prepared, the result is always just a “suggestion”, i.e. an assumption as to which gases the mixture might contain. This suggestion will have still to be examined, e.g. by considering the likelihood that a certain substance would be contained in the spectrum. In addition, recording a new spectrum for this substance can help to achieve clarity.

4.6.4 Quantitative gas analysis

Particular difficulties are encountered when interpreting the spectrum of an unknown mixture of gases. The proportions of ion flow from various sources can be offset one against the other only after all the sources have been identified. In many applications in vacuum technology one will be dealing with mixtures of a few simple gases of known identity, with atomic numbers less of than 50 (whereby the process-related gases can represent exceptions). In the normal, more complicated case there will be a spectrum with a multitude of superimpositions in a completely unknown mixture of many gas components; here a qualitative analysis will have to be made before attempting quantitative analysis. The degree of difficulty encountered will depend on the number of superimpositions (individual / a few / many).

In the case of individual superimpositions, mutual, balancing of the ion flows during measurement of one and the same type of gas for several atomic numbers can often be productive.

Where there is a larger number of superimpositions and a limited number of gases overall, tabular evaluation using correction factors vis à vis the spectrum of a calibration gas of known composition can often be helpful.

In the most general case a plurality of gases will make a greater or lesser

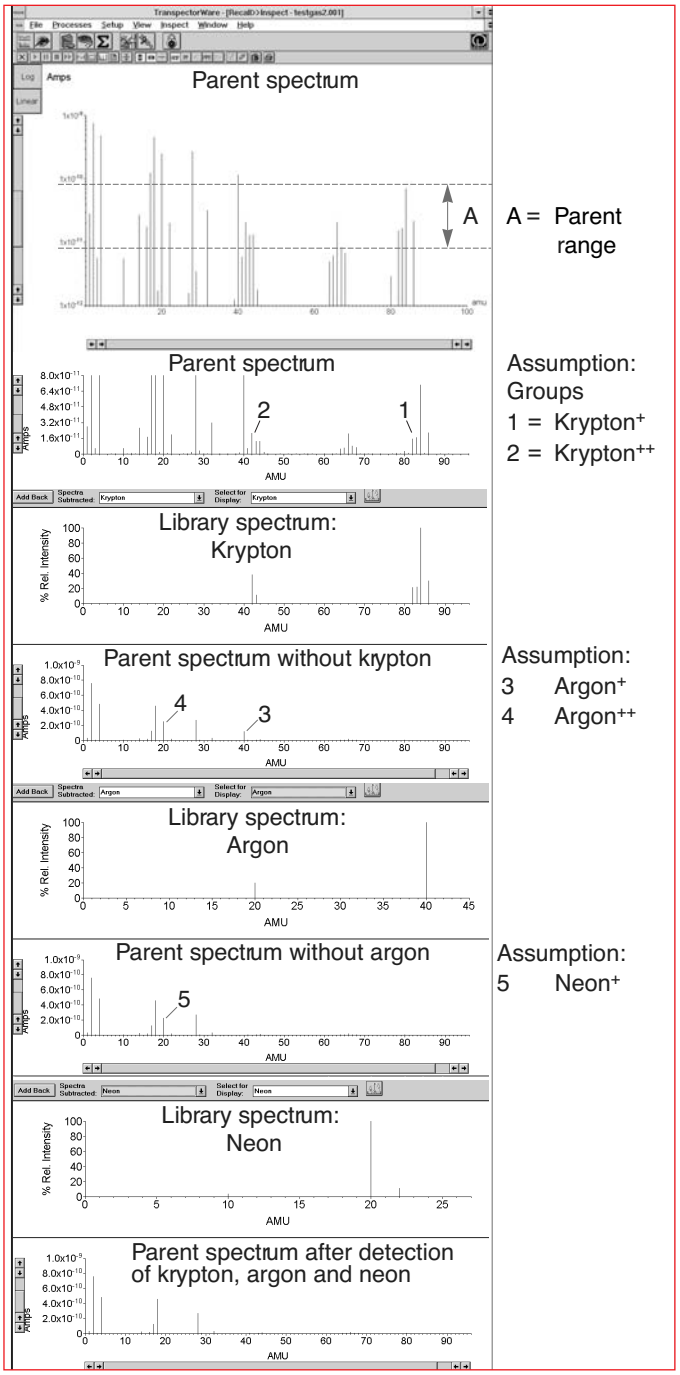


Fig. 4.16 Subtracting spectra contained in libraries

contribution to the ion flow for all the masses. The share of a gas g in each case for the atomic number m will be expressed by the fragment factor $F_{fm,g}$. In order to simplify calculation, the fragment factor $F_{fm,g}$ will also contain the transmission factor TF and the detection factor DF. Then the ion current to mass m, as a function of the overall ion currents of all the gases involved, in matrix notation, is:

$$\begin{bmatrix} i_j^+ \\ \cdot \\ \cdot \\ i_m^+ \\ \cdot \\ \cdot \\ i_u^+ \end{bmatrix} = \begin{bmatrix} BF_{j,k} & \cdot & \cdot & \cdot & \cdot & BF_{j,o} \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & FF_{m,g} & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ BF_{u,k} & \cdot & \cdot & \cdot & \cdot & BF_{u,o} \end{bmatrix} \cdot \begin{bmatrix} I_k^+ \\ \cdot \\ \cdot \\ I_g^+ \\ \cdot \\ \cdot \\ I_o^+ \end{bmatrix}$$

The ion current vector for the atomic numbers m (resulting from the contributions by the fragments of the individual gases) is equal to the fragment matrix times the vector of the sum of the flows for the individual gases.

or: $$i_m^+ = \sum_{g=k}^{0} BF_{m,g} \cdot I_g^+$$

(in simplified notation: $i = FF \cdot I$)

where i_m^+ = ion flow vector for the atomic numbers, resulting from contributions of fragments of various individual gases

$\sum_{g=k}^{0} BF_{m,g}$ = fragment matrix

I_g^+ = Vector of the sum of the flows for the individual gases or:

$$i_m^+ = \sum \overbrace{p_g \cdot E_{N_2} \cdot RIP_g \cdot FF_m \cdot TF_m}^{Ff_{m,g}}$$

- TF_m: Transmission factor for the mass m
- FF_m: Fragment factor for the gas to mass m
- RIP_g: Relative ionization probability for the gas
- E_{N_2}: Nitrogen sensitivity (equipment constant)
- p_g: Partial pressure of the gas
- i_m^+: Ion current for atomic number m

One sees that the ion flow caused by a gas is proportional to the partial pressure. The linear equation system can be solved only for the special instance where m = g (square matrix); it is over-identified for m > g. Due to unavoidable measurement error (noise, etc.) there is no set of overall ion flow I_g^+ (partial pressures or concentrations) which satisfies the equation system exactly. Among all the conceivable solutions it is now necessary to identify set I_g^{+*} which after inverse calculation to the partial ion flows I_m^{+*} will exhibit the smallest squared deviation from the partial ion currents i_m^+ actually measured. Thus:

$$\sum (i_m^+ - i_m^{+*})^2 = \min$$

This minimization problem is mathematically identical to the solution of another equation system

$$FF^T \cdot i = FF^T \cdot FF \cdot I$$

which can be evaluated direct by the computer. The ion current vector for the individual gases is then:

$$I = \frac{[FF^T \cdot i] \cdot [FF^T \cdot BF]^{-1}}{\det[FF^T \cdot BF]}$$

4.7 Software

4.7.1 Standard SQX software (DOS) for stand-alone operation (1 MS plus 1 PC, RS 232)

The conventional software package (SQX) contains the standard routines for the operation of the mass spectrometer (MS)– various spectra depictions, queries of individual channels with the corresponding screen displays as tables or bar charts, partial pressure conversion, trend displays, comparison with spectra libraries (with the capability for trial subtraction of library spectra), leak testing mode etc. – and for sensor balancing, as well. Using PCs as the computer and display unit naturally makes available all the usual functions including storing and retrieving data, printing, etc. Characteristic of the conventional software package is that specific individual spectra will be measured, even though the measurement is fully automated and takes place at a point in time which is specified in advance. A spectrum of this type can thus be only a “snapshot” of a process in progress.

4.7.2 Multiplex/DOS software MQX (1 to 8 MS plus 1 PC, RS 485)

The first step toward process-oriented software by LEYBOLD is the MQX. It makes possible simultaneous monitoring of a maximum of eight sensors and you can apply all the SQX functions at each sensor.

4.7.3 Process-oriented software – Transpector-Ware for Windows

Transpector-Ware is based on an entirely new philosophy. During the course of the process (and using settings – the “recipe” – determined beforehand) data will be recorded continuously – like the individual frames in a video. These data can be stored or otherwise evaluated. It is possible in particular to analyze interesting process sections exactly, both during the process and retroactively, once the process has run to completion, without having to interrupt the measurement operations which are running in the background. Where ongoing monitoring of identical processes is undertaken the program can generate statistics (calculating mean values and standard deviations) from which a bandwidth for “favorable process operation” can be derived. Error reports are issued where limit values are exceeded.

4.7.4 Development software – TranspectorView

This software used to for develop custom software versions for special situations. It is based on the LabView development package and includes the drivers required to operate the Transpector.

4.8 Partial pressure regulation

Some processes, such as reactive sputter processes, require the most constant possible incidence rates for the reacting gas molecules on the substrate being coated.

The “incidence rate” is the same as the “impingement rate” discussed in Chapter 1; it is directly proportional to the partial pressure. The simplest attempt to keep the partial pressure for a gas component constant is throughput by regulating with a flow controller; it does have the disadvantage that the regulator cannot determine whether, when and where the gas consumption or the composition of the gas in the vacuum chamber changes. The far superior and more effective option is partial pressure control using a mass spectrometer via gas inlet valves. Here the significant peaks of the gases being considered are assigned to channels in the mass spectrometer. Suitable regulators compare the analog output signals for these channels with set-point values and derive from the difference between the target and actual values for each channel the appropriate actuation signal for the gas inlet valve for the channel. A configuration of this kind has been realized to control six channels in the QUADREX PPC. Gas inlet valves matching the unit can also be delivered.

The gas used to measure the impingement rate (partial pressure) must naturally be drawn from a representative point in the vacuum chamber. When evaluating the time constant for a regulation circuit of this type it is important to take into account all the time aspects and not just the electrical signal propagation and the processing in the mass spectrometer, but also the vacuum-technology time constants and flow velocities, as illustrated in Figure 4.17. Pressure converters or unfavorably installed gas inlet lines joining the control valve and the vacuum vessel will make particularly large contributions to the overall time constant. It is generally better to establish a favorable S/N ratio with a large signal (i.e. through an inlet diaphragm with a large opening) rather than with long integration periods at the individual channels. Contrasted in Figure 4.18 are the effects of boosting pressure and lengthening the integration time on signal detectability. In depictions a, b and c only the integration period was raised, from 0.1 to 1.0 and 10 seconds (thus by an overall factor of 100), respectively. By comparison, in the sequence a-d-e-f, at constant integration time, the total pressure was raised in three steps, from 7.2 · 10^{-6} mbar to 7.2 · 10^{-5} mbar (or by a factor of just 10 overall).

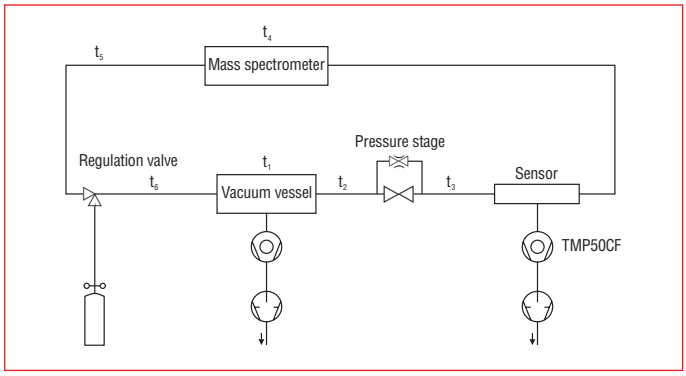


Fig. 4.17 Partial shares for overall time constants

4.9 Maintenance

(Cathode service life, sensor balancing, cleaning the ion source and rod system)

The **service life of the cathode** will depend greatly on the nature of the loading. Experience has shown that the product of operating period multiplied by the operating pressure can serve as a measure for the loading. Higher operating pressures (in a range of 1 · 10^{-4} to 1 · 10^{-3} mbar) have a particularly deleterious effect on service life, as do certain chemical influences such as refrigerants, for example. Changing out the cathode is quite easy, thanks to the simple design of the sensor. It is advisable, however, to take this opportunity to change out or at least clean the entire ion source.

Sensor balancing at the mass axis (often erroneously referred to as calibration) is done today in a very easy fashion with the software (e.g. SQX, Transpector-Ware) and can be observed directly in the screen. Naturally, not only the arrangement along the mass axis will be determined here, but also the shape of the lines, i.e. resolution and sensitivity (see Section 4.5).

It will be necessary to **clean the sensor** only in exceptional cases where it is heavily soiled. It is usually entirely sufficient to clean the ion source, which can be easily dismantled and cleaned. The rod system can be cleaned in an ultrasonic bath once it has been removed from the configuration. If dismantling the system is unavoidable due to particularly stubborn grime, then the adjustment of the rods which will be required afterwards will have to be carried out at the factory.

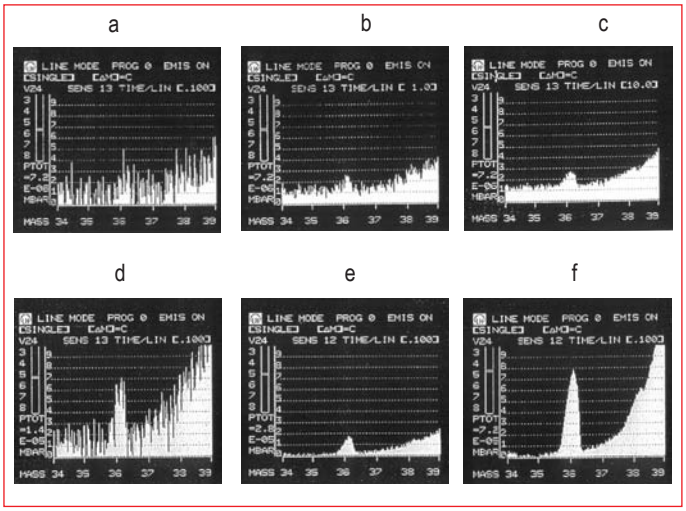


Fig. 4.18 Improving the signal-to-noise ratio by increasing the pressure or extending the integration time

Leak detection

5. Leaks and their detection

Apart from the vacuum systems themselves and the individual components used in their construction (vacuum chambers, piping, valves, detachable [flange] connections, measurement instruments, etc.), there are large numbers of other systems and products found in industry and research which must meet stringent requirements in regard to leaks or creating a so-called "hermetic" seal. Among these are many assemblies and processes in the automotive and refrigeration industries in particular but also in many other branches of industry. Working pressure in this case is often above ambient pressure. Here "hermetically sealed" is defined only as a relative "absence of leaks". Generalized statements often made, such as "no detectable leaks" or "leak rate zero", do not represent an adequate basis for acceptance testing. Every experienced engineer knows that properly formulated acceptance specifications will indicate a certain leak rate (see Section 5.2) under defined conditions. Which leak rate is acceptable is also determined by the application itself.

5.1 Types of leaks

Differentiation is made among the following leaks, depending on the **nature of the material or joining fault:**

- Leaks in detachable connections:
  Flanges, ground mating surfaces, covers
- Leaks in permanent connections:
  Solder and welding seams, glued joints
- Leaks due to porosity: particularly following mechanical deformation (bending!) or thermal processing of polycrystalline materials and cast components
- Thermal leaks (reversible): opening up at extreme temperature loading (heat/ cold), above all at solder joints
- Apparent (virtual) leaks: quantities of gas will be liberated from hollows and cavities inside cast parts, blind holes and joints (also due to the evaporation of liquids)
- Indirect leaks: leaking supply lines in vacuum systems or furnaces (water, compressed air, brine)
- "Serial leaks": this is the leak at the end of several "spaces connected in series", e.g. a leak in the oil-filled section of the oil pan in a rotary vane pump
- "One-way leaks": these will allow gas to pass in one direction but are tight in the other direction (very seldom)

An area which is not gas-tight but which is not leaky in the sense that a defect is present would be the

- Permeation (naturally permeability) of gas through materials such as rubber hoses, elastomer seals, etc. (unless these parts have become brittle and thus "leaky").

5.2 Leak rate, leak size, mass flow

No vacuum device or system can ever be absolutely vacuum-tight and it does not actually need to be. The simple essential is that the leak rate be low enough that the required operating pressure, gas balance and ultimate pressure in the vacuum container are not influenced. It follows that the requirements in regard to the gas-tightness of an apparatus are the more stringent the lower the required pressure level is. In order to be able to register leaks quantitatively, the concept of the "leak rate" with the symbol Q_L was introduced; it is measured with mbar · l/s or cm^3/s (STP) as the unit of measure. A leak rate of Q_L = 1 mbar · l/s is present when in an enclosed, evacuated vessel with a volume of 1 l the pressure rises by 1 mbar per second or, where there is positive pressure in the container, pressure drops by 1 mbar. The leak rate Q_L defined as a measure of leakiness is normally specified in the unit of measure mbar · l/s. With the assistance of the status equation (1.7) one can calculate Q_L when giving the temperature T and the type of gas M, registering this quantitatively as mass flow, e.g. in the g/s unit of measure. The appropriate relationship is then:

$$Q_L = \frac{\Delta(p \cdot V)}{\Delta t} = \frac{R \cdot T}{M} \cdot \frac{\Delta m}{\Delta t} \qquad (5.1)$$

where R = 83.14 mbar · l/mol · K, T = temperature in K; M = molar mass in g/mole; Δm for the mass in g; Δt is the time period in seconds. Equation 5.1 is then used

a) to determine the mass flow $\Delta m / \Delta t$ at a known pV gas flow of $\Delta p \cdot V/\Delta t$ (see in this context the example at 5.4.1) or

b) to determine the pV leak gas flow where the mass flow is known (see the following example).

Example for case b) above:

A refrigeration system using Freon (R 12) exhibits refrigerant loss of 1 g of Freon per year (at 25 °C). How large is the leak gas flow Q_L? According to equation 5.1 for M(R12) = 121 g/mole:

$$Q_L = \frac{\Delta(p \cdot V)}{\Delta t} = \frac{83.14\,\text{mbar} \cdot \ell \cdot 298\,\text{K} \cdot 1\,\text{g}}{\text{mol} \cdot \text{K} \cdot 121\,\text{g} \cdot \text{mol}^{-1} \cdot 1\,\text{year}}$$

$$= \frac{83.14 \cdot 2.98 \cdot 10^2 \cdot 1}{121 \cdot 1} \cdot \frac{\text{mbar} \cdot \ell}{3.15 \cdot 10^7\,\text{s}}$$

$$= \frac{83.14 \cdot 2.98 \cdot 10^2}{1.21 \cdot 10^2 \cdot 3.15} \cdot 10^{-7} \cdot \frac{\text{mbar} \cdot \ell}{\text{s}}$$

$$= 65 \cdot 10^{-7} \cdot \frac{\text{mbar} \cdot \ell}{\text{s}}$$

Thus the Freon loss comes to $Q_L = 6.5 \cdot 10^{-6}$ mbar · l/s. According to the "rule of thumb" for high vacuum systems given below, the refrigeration system mentioned in this example may be deemed to be very tight. Additional conversions for Q_L are shown in Tables VIIa and VIIb in Chapter 9.

The following rule of thumb for quantitative characterization of high vacuum equipment may be applied:

Total leak rate < 10^{-6} mbar · l/s:
Equipment is very tight

Total leak rate 10^{-5} mbar · l/s:
Equipment is sufficiently tight

Total leak rate > 10^{-4} mbar · l/s:
Equipment is leaky

A leak can in fact be “overcome” by a pump of sufficient capacity because it is true that (for example at ultimate pressure p_{end} and disregarding the gas liberated from the interior surfaces):

$$p_{end} = \frac{Q_L}{S_{eff}} \qquad (5.2)$$

(Q_L Leak rate, S_{eff} the effective pumping speed at the pressure vessel)

Where S_{eff} is sufficiently great it is possible – regardless of the value for the leak rate Q_L – always to achieve a pre-determined ultimate pressure of p_{end}. In practice, however, an infinite increase of S_{eff} will run up against economic and engineering limitations (such as the space required by the system).

Whenever it is not possible to achieve the desired ultimate pressure in an apparatus there are usually two causes which can be cited: The presence of leaks and/or gas being liberated from the container walls and sealants.

Partial pressure analysis using a mass spectrometer or the pressure rise method may be used to differentiate between these two causes. Since the pressure rise method will only prove the presence of a leak without indicating its location in the apparatus, it is advisable to use a helium leak detector with which leaks can, in general, also be located much more quickly.

In order to achieve an overview of the correlation between the geometric size of the hole and the associated leak rate it is possible to operate on the basis of the following, rough estimate: A circular hole 1 cm in diameter in the wall of a vacuum vessel is closed with a gate valve. Atmospheric pressure prevails outside, a vacuum inside. When the valve is suddenly opened all the air molecules in a cylinder 1 cm in diameter and 330 m high would within a 1-second period of time “fall into” the hole at the speed of sound (330 m/s). The quantity flowing into the vessel each second will be 10^{13} mbar times the cylinder volume (see Fig. 5.1). The result is that for a hole 1 cm in diameter Q_L (air) will be $2.6 \cdot 10^{4}$ mbar $\cdot$ l/s. If all other conditions are kept identical and helium is allowed to flow into the hole at its speed of sound of 970 m/s, then in analogous fashion the Q_L (helium) will come to $7.7 \cdot 10^{+4}$ mbar $\cdot$ l/s, or a pV leaking gas current which is larger by a factor of 970 / 330 = 2.94. This greater “sensitivity” for helium is used in leak detection practice and has resulted in the development and mass production of highly sensitive helium-based leak detectors (see Section 5.5.2).

Δp = 1013 mbar, Hole diameter d = 1 cm

Gas speed = Speed of sound = 330 $\frac{m}{s}$

Volume/second: $330 \frac{m}{s} \cdot \frac{1^2 \cdot \pi}{4}\ cm^2 = 25.95 \cdot 10^{+3}\ \frac{cm^3}{s} = 25.95\ \frac{\ell}{s}$

Quantity/second: $1013\ mbar \cdot 25.95\ \frac{\ell}{s} = 2.63 \cdot 10^{+4} \approx 10^{+4}\ \frac{mbar \cdot \ell}{s}$

Diameter cm	Leak rate $\frac{mbar \cdot \ell}{s}$
10^{-2} m = 1.0 cm	10^{+4}
10^{-3} m = 1.0 mm	10^{+2}
10^{-4} m = 0.1 mm	10^{0} (= 1)
10^{-5} m = 0.01 mm	10^{-2}
10^{-6} m = 1.0 µm	10^{-4}
10^{-7} m = 0.1 µm	10^{-6}
10^{-8} m = 0.01 µm	10^{-8}
10^{-9} m = 1.0 nm	10^{-10}
10^{-10} m = 1.0 Angstrom	10^{-12} (Detection limit, He leak detector)

Fig. 5.1 Correlation between leak rate and hole size

Shown in Figure 5.1 is the correlation between the leak rate and hole size for air, with the approximation value of Q_L (air) of 10^{+4} mbar $\cdot$ l/s for the “1 cm hole”. The table shows that when the hole diameter is reduced to 1 µm (= 0.001 mm) the leak rate will come to 10^{-4} mbar $\cdot$ l/s, a value which in vacuum technology already represents a major leak (see the rule of thumb above). A leak rate of 10^{-12} mbar $\cdot$ l/s corresponds to hole diameter of 1 Å; this is the lower detection limit for modern helium leak detectors. Since the grid constants for many solids amount to several Å and the diameter of smaller molecules and atoms (H_2, He) are about 1 Å, inherent permeation by solids can be registered metrologically using helium leak detectors. This has led to the development of calibrated reference leaks with very small leak rates (see Section 5.5.2.3). This is a measurable “lack of tightness” but not a “leak” in the sense of being a defect in the material or joint. Estimates or measurements of the sizes of atoms, molecules, viruses, bacteria, etc. have often given rise to everyday terms such as “watertight” or “bacteria-tight”; see Table 5.1.

Compiled in Figure 5.2 are the nature and detection limits of frequently used leak detection methods.

Concept / criterion	Comment	Q_L [mbar $\cdot$ l/s]	Relevant particle size
Water-tight*)	Droplets	$Q_L < 10^{-2}$	
Vapor-tight	“Sweating”	$Q_L < 10^{-3}$	
Bacteria-tight*)			
(cocci)		$Q_L < 10^{-4}$	Avg. ≈ 1 µm
(rod-shaped)			Avg. ≈ 0.5-1 µm, 2–10 µm long
Oil-tight		$Q_L < 10^{-5}$	
Virus-tight*)			
(vaccines such as pox)		$Q_L < 10^{-6}$	Ø ≈ $3 \cdot 10^{-7}$ m
(smallest viruses,			
bacteriophages)		$Q_L < 10^{-8}$	Ø ≈ $3 \cdot 10^{-8}$ m
(viroids, RNA)		$Q_L < 10^{-10}$	Ø [a] ≈ $1 \cdot 10^{-9}$ m (thread-like)
Gas-tight		$Q_L < 10^{-7}$	
“Absolutely tight”	Technical	$Q_L < 10^{-10}$	

*) As opposed to vapor, it is necessary to differentiate between hydrophilic and hydrophobic solids. This also applies to bacteria and viruses since they are transported primarily in solutions.

Table 5.1 Estimating borderline leak rates

Leak detection

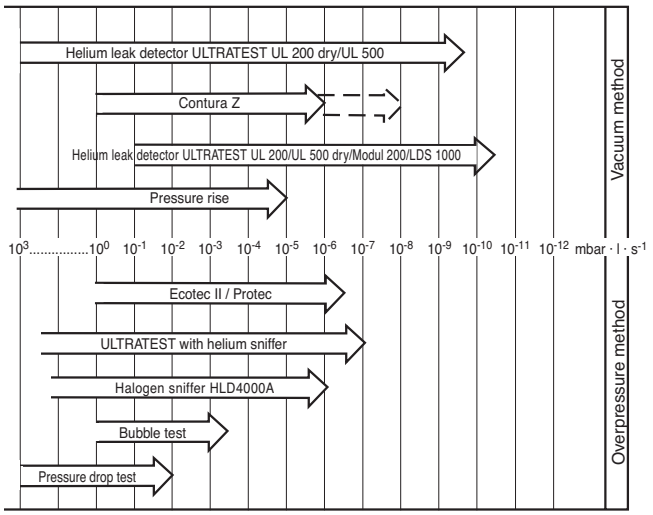


Fig. 5.2 Leak rate ranges for various leak detection processes and devices

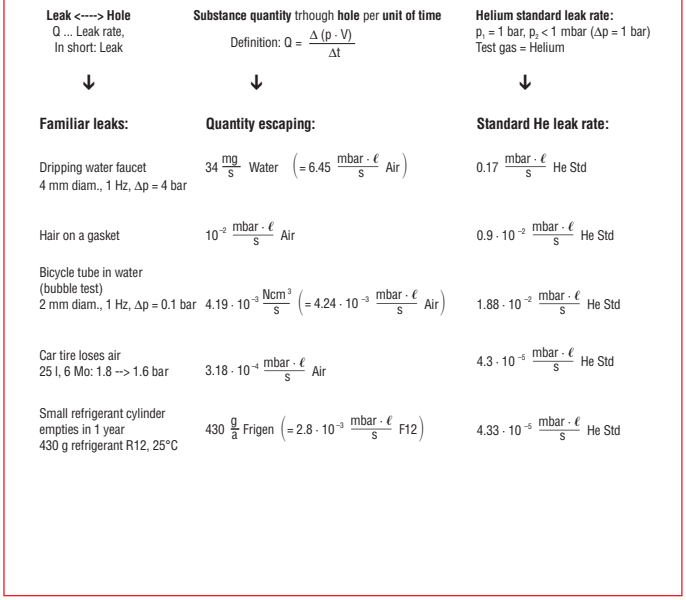


Fig. 5.3 Examples for conversion into helium standard leak rates

5.2.1 The standard helium leak rate

Required for unequivocal definition of a leak are, first, specifications for the pressures prevailing on either side of the partition and, secondly, the nature of the medium passing through that partition (viscosity) or its molar mass. The designation “helium standard leak” (He Std) has become customary to designate a situation frequently found in practice, where testing is carried out using helium at 1 bar differential between (external) atmospheric pressure and the vacuum inside a system (internal, p < 1 mbar), the designation “helium standard leak rate” has become customary. In order to indicate the rejection rate for a test using helium under standard helium conditions it is necessary first to convert the real conditions of use to helium standard conditions (see Section 5.2.2). Some examples of such conversions are shown in Figure 5.3.

5.2.2 Conversion equations

When calculating pressure relationships and types of gas (viscosity) it is necessary to keep in mind that different equations are applicable to laminar and molecular flow; the boundary between these areas is very difficult to ascertain. As a guideline one may assume that laminar flow is present at leak rates where $Q_L > 10^{-5}$ mbar · l/s and molecular flow at leak rates where $Q_L < 10^{-7}$ mbar · l/s. In the intermediate range the manufacturer (who is liable under the guarantee terms) must assume values on the safe side. The equations are listed in Table 5.2.

Range	Laminar	Molecular
Pressure		
Gas	$Q_{gas\ A} \cdot \eta_{gas\ A} = Q_{gas\ B} \cdot \eta_{gas\ B}$	

Table 5.2 Conversion formulae for changes of pressure and gas type

Here indices “I” and “II” refer to the one or the other pressure ratio and indices “1” and “2” reference the inside and outside of the leak point, respectively.

5.3 Terms and definitions

When searching for leaks one will generally have to distinguish between two tasks:

1. Locating leaks and
2. Measuring the leak rate.

In addition, we distinguish, based on the direction of flow for the fluid, between the

a. **vacuum method** (sometimes known as an “outside-in leak”), where the direction of flow is into the test specimen (pressure inside the specimen being less than ambient pressure), and the

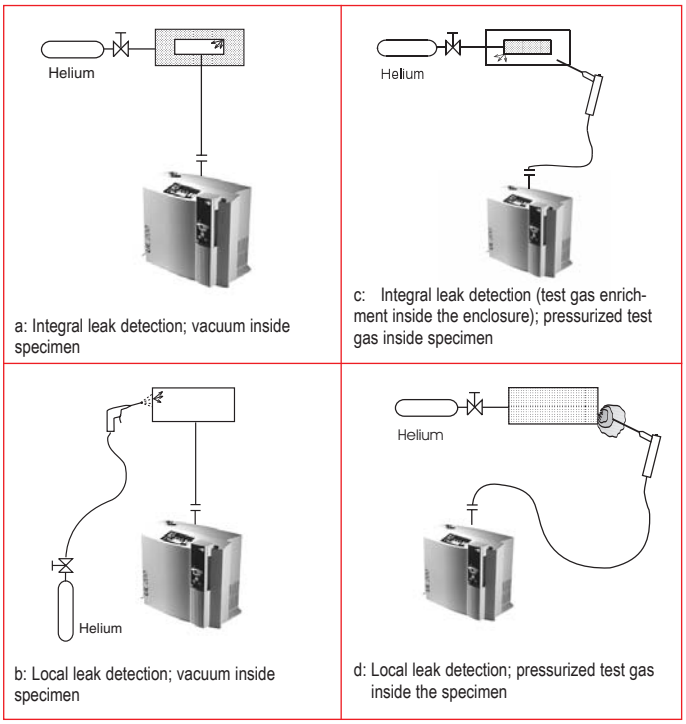
b. **positive pressure method** (often referred to as the “inside-out leak”), where the fluid passes from inside the test specimen outward (pressure inside the specimen being greater than ambient pressure).

The specimens should wherever possible be examined in a configuration corresponding to their later application – components for vacuum applications using the vacuum method and using the positive pressure method for parts which will be pressurized on the inside.

When measuring leak rates we differentiate between registering

a. individual leaks (**local measurement**) – sketches b and d in Figure 5.4, and registering

b. the total of all leaks in the test specimen (**integral measurement**) – sketches a and c in Figure 5.4.



a: Integral leak detection; vacuum inside specimen

c: Integral leak detection (test gas enrichment inside the enclosure); pressurized test gas inside specimen

b: Local leak detection; vacuum inside specimen

d: Local leak detection; pressurized test gas inside the specimen

Fig. 5.4 Leak test techniques and terminology

The leak rate which is no longer tolerable in accordance with the acceptance specifications is known as the **rejection rate**. Its calculation is based on the condition that the test specimen may not fail during its planned utilization period due to faults caused by leaks, and this to a certain degree of certainty. Often it is not the leak rate for the test specimen under normal operating conditions which is determined, but rather the throughput rate of a test gas – primarily helium – under test conditions. The values thus found will have to be converted to correspond to the actual application situation in regard to the pressures inside and outside the test specimen and the type of gas (or liquid) being handled.

Where a vacuum is present inside the test specimen ($p < 1$ mbar), atmospheric pressure outside, and helium is used at the test gas, one refers to **standard helium conditions**. Standard helium conditions are always present during helium leak detection for a high vacuum system when the system is connected to a leak detector and is sprayed with helium (**spray technique**). If the specimen is evacuated solely by the leak detector, then one would say that the leak detector is operating in the **direct-flow mode**. If the specimen is itself a complete vacuum system with its own vacuum pump and if the leak detector is operated in parallel to the system's pumps, then one refers to **partial-flow mode**. One also refers to partial stream mode when a separate auxiliary pump is used parallel to the leak detector.

When using the positive pressure method it is sometimes either impractical or in fact impossible to measure the leakage rate directly while it could certainly be sensed in an envelope which encloses the test specimen. The measurement can be made by connecting that envelope to the leak detector or by accumulation (increasing the concentration) of the test gas inside the envelope. The "**bombing test**" is a special version of the accumulation test (see Section 5.7.4). In the so-called **sniffer technique**, another variation of the of the positive pressure technique, the (test) gas issuing from leaks is collected (extracted) by a special apparatus and fed to the leak detector. This procedure can be carried out using either helium or refrigerants or SF_6 as the test gas.

5.4 Leak detection methods without a leak detector unit

The most sensible differentiation between the test methods used is differentiation as to whether or not special leak detection equipment is used.

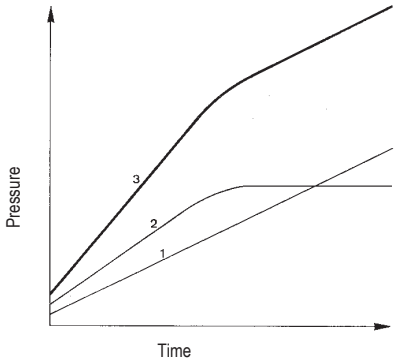
In the simplest case a leak can be determined qualitatively and, when using certain test techniques, quantitatively as well (this being the leak rate) without the assistance of a special leak detector. Thus the quantity of water dripping from a leaking water faucet can be determined, through a certain period of time, using a graduated cylinder but one would hardly refer to this as a leak detector unit. In those cases where the leak rate can be determined during the course of the search for the leak without using a leak detector (see, for example, Sect. 5.4.1), this will often be converted to the helium standard leak rate (Sect. 5.2.1). This standard leak rate value is frequently needed when issuing acceptance certificates but can also be of service when comparing leak rate values determined by helium leak detector devices.

In spite of careful inspection of the individual engineering components, leaks may also be present in an apparatus following its assembly – be it due to poorly seated seals or damaged sealing surfaces. The processes used to examine an apparatus will depend on the size of the leaks and on the degree of tightness being targeted and also on whether the apparatus is made of metal or glass or other materials. Some leak detection techniques are sketched out below. They will be selected for use in accordance with the particular application situations; economic factors may play an important part here.

5.4.1 Pressure rise test

This leak testing method capitalizes on the fact that a leak will allow a quantity of gas – remaining uniform through a period of time – to enter a sufficiently evacuated device (impeded gas flow, see Fig. 1.1). In contrast, the quantity of gas liberated from container walls and from the materials used for sealing (if these are not sufficiently free of outgassing) will decline through time since these will practically always be condensable vapors for which an equilibrium pressure is reached at some time (see Fig. 5.5). The valve at the pump end of the evacuated vacuum vessel will be closed in preparation for pressure rise measurements. Then the time is measured during which the pressure rises by a certain amount (by one power of ten, for example). The valve is opened again and the pump is run again for some time, following which the process will be repeated. If the time noted for this same amount of pressure rise remains constant, then a leak is present, assuming that the waiting period between the two pressure rise trials was long enough. The length of an appropriate waiting period will depend on the nature and size of the device. If the pressure rise is more moderate during the second phase, then the rise may be assumed to result from gases liberated from the inner surfaces of the vessel. One may also attempt to differentiate between leaks and contamination by interpreting the curve depicting the rise in pressure. Plotted on a graph with linear scales, the curve for the rise in pressure must be a straight line where a leak is present, even at higher pressures. If the pressure rise is due to gas being liberated from the walls (owing ultimately to contamination), then the pressure rise will gradually taper off and will approach a final and stable value. In most cases both phenomena will occur simultaneously so that separating the two causes is often difficult if not impossible. These relationships are

Leak detection



1 Leak
2 Gas evolved from the container walls
3 Leak + gas evolution

Fig. 5.5 Pressure rise within a vessel after the pump is switched off

shown schematically in Figure 5.5. Once it has become clear that the rise in pressure is due solely to a real leak, then the leak rate can be determined quantitatively from the pressure rise, plotted against time, in accordance with the following equation:

$$Q_L = \frac{\Delta p \cdot V}{\Delta t} \qquad (5.3)$$

Example: Once the vacuum vessel with a volume of 20 l has been isolated from the pump, the pressure in the apparatus rises from $1 \cdot 10^{-4}$ mbar to $1 \cdot 10^{-3}$ mbar in 300 s. Thus, in accordance with equation 5.2, the leak rate will be

$$Q_L = \frac{\left(1 \cdot 10^{-3} - 1 \cdot 10^{-4}\right) \cdot 20}{300}$$

$$= \frac{9 \cdot 10^{-4} \cdot 20}{300} = 6 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}}$$

The leak rate, expressed as mass flow $\Delta m / \Delta t$, is derived from equation 5.1 at $Q_L = 6 \cdot 10^{-5}$ mbar · l/s, T = 20 °C and the molar mass for air (M = 29 g/mole) at

$$Q_L = \frac{\Delta m}{\Delta t} = 6 \cdot 10^{-5} \cdot \frac{\text{mbar} \cdot \ell}{\text{s}} \cdot 29 \frac{\text{g}}{\text{mol}} \cdot$$

$$\cdot \frac{\text{mol} \cdot \text{K}}{83.14\,\text{mbar} \cdot \ell \cdot 293 \cdot 10^2\,\text{K}} = 7 \cdot 10^{-8} \frac{\text{g}}{\text{s}}$$

If the container is evacuated with a TURBOVAC 50 turbomolecular pump, for example (S = 50 l/s), which is attached to the vacuum vessel by way of a shut-off valve, then one may expect an effective pumping speed of about S_{eff} = 30 l/s. Thus the ultimate pressure will be

$$p_{end} = \frac{Q_L}{S_{eff}} = \frac{6 \cdot 10^{-5}\,\text{mbar} \cdot \ell \cdot \text{s}^{-1}}{30 \ell \cdot \text{s}^{-1}}$$

$$= 2 \cdot 10^{-6}\,\text{mbar}$$

Naturally it is possible to improve this ultimate pressure, should it be insufficient, by using a larger-capacity pump (e.g. the TURBOVAC 151) and at the same time to reduce the pump-down time required to reach ultimate pressure.

Today leak tests for vacuum systems are usually carried out with helium leak detectors and the vacuum method (see Section 5.7.1). The apparatus is evacuated and a test gas is sprayed around the outside. In this case it must be possible to detect (on the basis of samplings inside the apparatus) the test gas which has passed through leaks and into the apparatus. Another option is to use the positive-pressure leak test. A test gas (helium) is used to fill the apparatus being inspected and to build up a slight positive pressure; the test gas will pass to the outside through the leaks and will be detected outside the device. The leaks are located with leak sprays (or soap suds, 5.4.5) or – when using He or H_2 as the test gas – with a leak detector and sniffer unit (5.7.2).

5.4.2 Pressure drop test

The thinking here is analogous to that for the pressure rise method (Section 5.4.1). The method is, however, used only rarely to check for leaks in vacuum systems. If this is nonetheless done, then gauge pressure should not exceed 1 bar since the flange connectors used in vacuum technology will as a rule not tolerate higher pressures. Positive pressure testing is, on the other hand, a technique commonly employed in tank engineering. When dealing with large containers and the long test periods they require for the pressure drop there it may under certain circumstances be necessary to consider the effects of temperature changes. As a consequence it may happen, for example, that the system cools to below the saturation pressure for water vapor, causing water to condense; this will have to be taken into account when assessing the pressure decline.

5.4.3 Leak test using vacuum gauges which are sensitive to the type of gas

The fact that the pressure reading at vacuum gauges (see Section 3.3) is sensitive to the type of gas involved can, to a certain extent, be utilized for leak detection purposes. Thus it is possible to brush or spray suspected leaks with alcohol. The alcohol vapors which flow into the device – the thermal conductivity and ionizablity of which will vary greatly from the same properties for air – will affect and change pressure indication to a greater or lesser extent. The availability of more precise, easy-to-use helium leak detectors has, however, rendered this method almost completely obsolete.

5.4.4 Bubble immersion test

The pressurized test specimen is submerged in a liquid bath. Rising gas bubbles indicate the leak. Leak detection will depend greatly on the attentiveness of the person conducting the test and involves the temptation to increase the "sensitivity" by using ever higher temperatures, wherein the applicable safety regulations are sometimes disregarded. This method is very time-consuming for smaller leaks, as Table 5.3 shows. It references leak testing on a refrigeration system using type R12 refrigerant. Here the leak rate is specified in grams of refrigerant loss per year (g/a). Water is used as a test liquid (which may be heated or to which a surfactant may be added) or petroleum-based oils. The surface tension should not exceed 75 dyn/cm (1 dyn = 10^{-5} N).

Freon F12 loss per year (g/a)	Time taken to form a gas bubble (s)	Equivalent leak rate (cm^3[STP]/s)	Detection time using helium leak detector (s)
280	13.3	$1.8 \cdot 10^{-3}$	a few seconds
84	40	$5.4 \cdot 10^{-4}$	a few seconds
28	145	$1.8 \cdot 10^{-4}$	a few seconds
14	290	$9.0 \cdot 10^{-5}$	a few seconds
2.8	24 min	$1.8 \cdot 10^{-5}$	a few seconds
0.28 *	6 h	$1.8 \cdot 10^{-6}$	a few seconds

*) This leak rate represents the detection limit for good halogen leak detectors (≈ 0,1 g/a).

Table 5.3 Comparison of bubble test method (immersion technique) wit helium leak detector

5.4.5 Foam-spray test

In many cases pressurized containers or gas lines (including the gas supply lines for vacuum systems) can be checked quite conveniently for leaks by brushing or spraying a surfactant solution on them. Corresponding leak detection sprays are also available commercially. Escaping gas forms "soap bubbles" at the leak points. Here, again, the detection of smaller leaks is time-consuming and will depend greatly on the attentiveness of the inspector. The hydrogen gas cooling systems used in power plant generators represent a special case. These are indeed sometimes tested in the fashion described above but they can be examined much better and at much higher sensitivity by "sniffing" the hydrogen escaping at leaks using a helium leak detector which has been adjusted to respond to H2 (see Section 5.7.2).

5.4.6 Vacuum box check bubble

As a variation on the spray technique mentioned above, in which the escaping gas causes the bubbles, it is possible to place a so-called "vacuum box" with a seal (something like a diver's goggles) on the surface being examined once it has been sprayed with a soap solution. This box is then evacuated with a vacuum pump. Air entering from the outside through leaks will cause bubbles inside the box, which can be observed through a glass window in the box. In this way it is also possible, for example, to examine flat sheet metal plates for leaks. Vacuum boxes are available for a variety of applications, made to suit a wide range of surface contours.

5.4.7 Krypton 85 test

When dealing with small, hermetically sealed parts where the enclosure is leaky, krypton 85, a gaseous, radioactive isotope, can first be forced into the device by applying pressure from the outside. Once an exactly measured holding period has elapsed the pressure will be relieved, the component flushed and the activity of the "gas charge" will be measured. In the same way it is also possible to use helium as the test gas (see Section 5.7.4, bombing test).

5.4.8 High-frequency vacuum test

The so-called high-frequency vacuum tester can be used not only to check the pressure in glass equipment but also to locate porous areas in plastic or paint coatings on metals. This comprises a hand-held unit with a brush-like high-frequency electrode and a power pack. The shape and color of the electrical gas discharge can serve as a rough indicator for the pressure prevailing inside glass equipment. In the case of the vacuum tester – which comprises primarily a tesla transformer (which delivers a high-voltage, high-frequency AC current) – the corona electrode approaching the apparatus will trigger an electrode-free discharge inside the apparatus. The intensity and color of this discharge will depend on the pressure and the type of gas. The luminous discharge phenomenon allows us to draw conclusions regarding the approximate value for the pressure prevailing inside the apparatus. The discharge luminosity will disappear at high and low pressures.

When searching for leaks in glass equipment the suspect sections will be scanned or traced with the high-frequency vacuum tester electrode. Where there is a leak an arc will strike through to the pore in the glass wall, tracing a brightly lit discharge trail. Small pores can be enlarged by these sparks! The corona discharge of the vacuum tester can also penetrate thin areas in the glass particularly at weld points and transitional areas between intermediate components. Equipment which was originally leak-free can become leaky in this fashion! In contrast to the actual leak detector units, the high-frequency vacuum tester is highly limited in its functioning.

5.4.9 Test with chemical reactions and dye penetration

Occasionally leaks can also be located or detected by means of chemical reactions which result in a discoloration or by penetration of a dye solution into fine openings. The discoloration of a flame due to halogen gas escaping through leaks was used earlier to locate leaks in solder joints for refrigeration units.

A less frequently employed example of a chemical effect would be that of escaping ammonia when it makes contact with ozalid paper (blueprint paper) or with other materials suitably prepared and wrapped around the outside of the specimen. Leaks are then detected based on the discoloration of the paper.

An example of a dye penetration test is the inspection of the tightness of rubber plugs or plungers in glass tubes, used for example in testing materials suitability for disposable syringes or pharmaceutical packages. When evaluating tiny leaks for liquids it will be necessary to consider the wetability of the surface of the solid and the capillary action; see also Table 5.1.

Leak detection

Some widely used leak detection methods are shown – together with the test gas, application range and their particular features – in Table 5.4.

Method	Test gas	Smallest detectable leak rate		Pressure range	Quantitative measurement
		mbar ℓ/s	g/a R 134 a		
Foaming liquids	Air and others	10^{-4}	$7 \cdot 10^{-1}$	Positive pressure	No
Ultrasonic microphone	Air and others	10^{-2}	70	Positive pressure	No
Thermal conductivity leak detector	Gases other than air	$10^{-3} – 10^{-5}$	$10^{-1} – 7$	Positive pressure and vacuum	No
Halogen leak detection	Substances containing halogens	10^{-6} (10^{-5})	$7 \cdot 10^{-3}$ (10^{-1})	Positive pressure (vacuum)	With limitations
Universal sniffer leak detector	Refrigerants, helium and other gases	10^{-5}	$7 \cdot 10^{-3}$	Positive pressure	Yes
Helium leak detection	Helium	10^{-12} 10^{-7}	$7 \cdot 10^{-9}$ $7 \cdot 10^{-4}$	Vacuum, positive pressure	Yes
Bubble test	Air and other gases	10^{-3}	7	Positive pressure	No
Water pressure test	Water	10^{-2}	70	Positive pressure	No
Pressure drop test	Air and other gases	10^{-4}	$7 \cdot 10^{-1}$	Positive pressure	Yes
Pressure rise test	Air	10^{-4}	$7 \cdot 10^{-1}$	Vacuum	Yes

Table 5.4 Comparison of leak detection methods

5.5 Leak detectors and how they work

Most leak testing today is carried out using special leak detection devices. These can detect far smaller leak rates than techniques which do not use special equipment. These methods are all based on using specific gases for testing purposes. The differences in the physical properties of these test gases and the gases used in real-life applications or those surrounding the test configuration will be measured by the leak detectors. This could, for example, be the differing thermal conductivity of the test gas and surrounding air. The most widely used method today, however, is the detection of helium used as the test gas.

The function of most leak detectors is based on the fact that testing is conducted with a special test gas, i.e. with a medium other than the one used in normal operation. The leak test may, for example, be carried out using helium, which is detected using a mass spectrometer, even though the component being tested might, for example, be a cardiac pacemaker whose interior components are to be protected against the ingress of bodily fluids during normal operation. This example alone makes it clear that the varying flow properties of the test and the working media need to be taken into consideration.

5.5.1 Halogen leak detectors (HLD 4000, D-Tek)

Gaseous chemical compounds whose molecules contain chlorine and/or fluorine – such as refrigerants R12, R22 and R134a – will influence the emissions of alkali ions from a surface impregnated with a mixture of KOH and Iron(III)hydroxide and maintained at 800 °C to 900 °C by an external Pt heater. The released ions flow to a cathode where the ion current is measured and then amplified (halogen diode principle). This effect is so great that partial pressures for halogens can be measured down to 10^{-7} mbar.

Whereas such devices were used in the past for leak testing in accordance with the vacuum method, today – because of the problems associated with the CFCs – more sniffer units are being built. The attainable detection limit is about $1 \cdot 10^{-6}$ mbar $\cdot$ l/s for all the devisces. Equipment operating in accordance with the halogen diode principle can also detect SF_6. Consequently these sniffer units are used to determine whether refrigerants are escaping from a refrigeration unit or from an SF_6 type switch box (filled with arc suppression gas).

5.5.2 Leak detectors with mass spectrometers (MSLD)

The detection of a test gas using mass spectrometers is far and away the most sensitive leak detection method and the one most widely used in industry. The MS leak detectors developed for this purpose make possible quantitative measurement of leak rates in a range extending across many powers of ten (see Section 5.2) whereby the lower limit $\approx 10^{-12}$ mbar $\cdot$ l/s, thus making it possible to demonstrate the inherent gas permeability of solids where helium is used as the test gas. It is actually possible in principle to detect all gases using mass spectrometry. Of all the available options, the use of helium as a tracer gas has proved to be especially practical. The detection of helium using the mass spectrometer is absolutely (!) unequivocal. Helium is chemically inert, non-explosive, non-toxic, is present in normal air in a concentration of only 5 ppm and is quite economical. Two types of mass spectrometer are used in commercially available MSLD's:

a) The quadrupole mass spectrometer, although this is used less frequently due to the more elaborate and complex design (above all due to the electrical supply for the sensor), or

b) the 180° magnetic sector field mass spectrometer, primarily due to the relatively simple design.

Regardless of the functional principle employed, every mass spectrometer comprises three physically important sub-systems: the ion source, separation system and ion trap. The ions must be able to travel along the path from the ion source and through the separation system to the ion trap, to the greatest possible extent without colliding with gas molecules. This path amounts to about 15 cm for all types of spectrometers and thus requires a medium free path length of at least 60 cm, corresponding to pressure of about $1 \cdot 10^{-4}$ mbar; in other words, a mass spectrometer will operate only in a vacuum. Due to the minimum vacuum level of $1 \cdot 10^{-4}$ mbar, a high vacuum will be required. Turbomolecular pumps and suitable roughing pumps are used in modern leak detectors. Associated with the individual component groups are the required electrical- and electronic supply systems and software which, via a microprocessor, allow for the greatest pos-

sible degree of automation in the operating sequence, including all adjustment and calibration routines and measured value display.

5.5.2.1 The operating principle for a MSLD

The basic function of a leak detector and the difference between a leak detector and mass spectrometer can be explained using Figure 5.6. This sketch shows the most commonly found configuration for leak detection using the helium spray method (see Section 5.7.1) at a vacuum component. When the sprayed helium is drawn into the component through a leak it is pumped thorough the interior of the leak detector to the exhaust, where it again leaves the detector. Assuming that the detector itself is free of leaks, the amount of gas flowing through each pipe section (at any desired point) per unit of time will remain constant regardless of the cross section and the routing of the piping. The following applies for the entry into the pumping port at the vacuum pump:

$$Q = p \cdot S \qquad (5.4)$$

At all other points

$$Q = p \cdot S_{eff} \qquad (5.4a)$$

applies, taking the line losses into account.

The equation applies to all gases which are pumped through the piping and thus also for helium.

$$Q_{He} = p_{He} \cdot S_{eff,\,He} \qquad (5.4b)$$

In this case the gas quantity per unit of time is the leak rate being sought; the total pressure may not be used, but only the share for helium or the partial pressure for helium. This signal is delivered by the mass spectrometer when it is set for atomic number 4 (helium). The value for S_{eff} is a constant for every series of leak detectors, making it possible to use a microprocessor to multiply the signal arriving from the mass spectrometer by a numerical constant and to have the **leak rate displayed direct**.

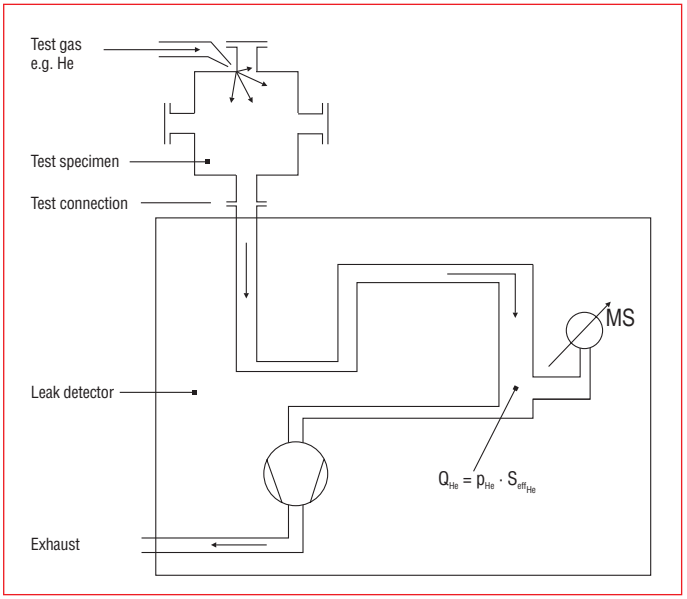


Fig. 5.6 Basic operating principle for a leak detector

5.5.2.2 Detection limit, background, gas storage in oil (gas ballast), floating zero-point suppression

The smallest detectable leak rate is dictated by the natural background level for the gas to be detected. Even with the test connector at the leak detector closed, every gas will pass – counter to the pumping direction – through the exhaust and through the pumps (but will be reduced accordingly by their compression) through to the spectrometer and will be detected there if the electronic means are adequate to do so. The signal generated represents the detection limit. The high vacuum system used to evacuate the mass spectrometer will normally comprise a turbomolecular pump and an oil-sealed rotary vane pump. (Diffusion pumps were used earlier instead of the turbomolecular pumps.) Like every liquid, the sealing oil in the rotary vane pump has the capability of dissolving gases until equilibrium is reached between the gas dissolved in the oil and the gas outside the oil. When the pump is warm (operating temperature) this equilibrium state represents the detection limit for the leak detector. The helium stored in the oil thus influences the detection limit for the leak detector. It is possible for test gas to enter not only through the test connection and into the leak detector; improper installation or inept handling of the test gas can allow test gas to enter through the exhaust and the airing or gas ballast valve and into the interior of the detector, to increase the helium level in the oil and the elastomer seals there and thus to induce a background signal in the mass spectrometer which is well above the normal detection limit. When the device is properly installed (see Fig. 5.7) the gas ballast valve and the airing valve will be connected to fresh air and the discharge line (oil filter!) should at least be routed to outside the room where the leak test takes place.

An increased test gas (helium) background level can be lowered by opening the gas ballast valve and introducing gas which is free of the test gas (helium-free gas, fresh air). The dissolved helium will be flushed out, so to speak. Since the effect always affects only the part of the oil present in the pump body at the particular moment, the flushing procedure will have to be continued until all the oil from the pump's oil pan has been recirculated several times. This period of time will usually be 20 to 30 minutes.

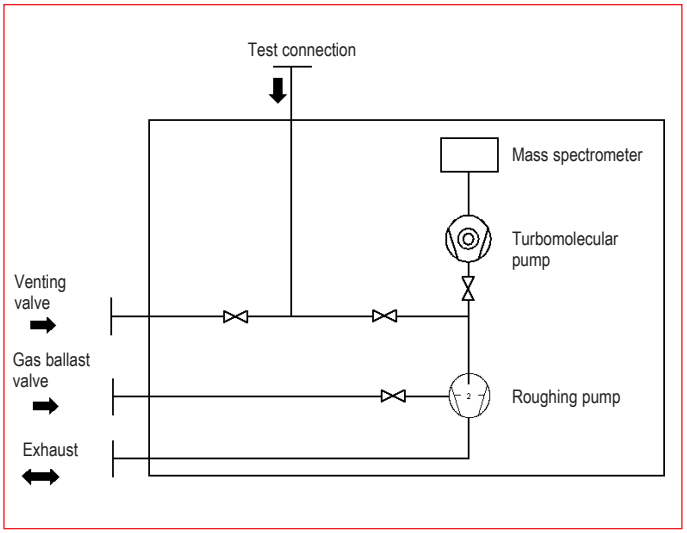


Fig. 5.7 Correct set-up for a MSLD

Leak detection

In order to spare the user the trouble of always having to keep an eye on the background level, what has been dubbed **floating zero-point suppression** has been integrated into the automatic operating concepts of all INFICON leak detectors (Section 5.5.2.5). Here the background level measured after the inlet valve has been closed is placed in storage; when the valve is then opened again this value will automatically be deducted from subsequent measurements. Only at a relatively high threshold level will the display panel show a warning indicating that the background noise level is too high. Figure 5.8 is provided to illustrate the process followed in zero point suppression. Chart on the left. The signal is clearly larger than the background. Center chart: the background has risen considerably; the signal can hardly be discerned. Chart on the right: the background is suppressed electrically; the signal can again be clearly identified.

Independent of this floating zero-point suppression, all the leak detectors offer the capability for manual zero point shifting. Here the display for the leak detector at the particular moment will be "reset to zero" so that only rises in the leak rate from that point on will be shown. This serves only to facilitate the evaluation of a display but can, of course, not influence its accuracy.

Modern leak detectors are being more frequently equipped with oil-free vacuum systems, the so-called "dry leak detectors" (UL 200 dry, UL 500 dry). Here the problem of gas being dissolved in oil does not occur but similar purging techniques will nonetheless be employed.

5.5.2.3 Calibrating leak detectors; test leaks

Calibrating a leak detector is to be understood as matching the display at a leak detector unit, to which a test leak is attached, with the value shown on the "label" or calibration certificate. The prerequisite for this is correct adjustment of the ion paths in the spectrometer, also known as tuning. Often the distinction is not made quite so carefully and both procedures together are referred to as calibration.

In the calibration process proper the straight-line curve representing the numerically correct, linear correlation between the gas flow per unit of time and the leak rate is defined by two points: the zero point (no display where no emissions are detected) and the value shown with the test leak (correct display for a known leak).

In vacuum operations (spray technique, see Section 7.1) one must differentiate between two types of calibration: with an internal or external test leak. When using a test leak built into the leak detector the unit can itself be calibrated but it can only calibrate itself. When using an external test leak not just the device but also a complete configuration, such as a partial flow arrangement, can be included. Internal test leaks are permanently installed and cannot be misplaced. At present all the leak detectors being distributed by INFICON are fitted with an automatic calibration routine.

Sniffer units or configurations will as a rule have to be calibrated with special, external test leaks in which there is a guarantee that on the one hand all the test gas issuing from the test leak reaches the tip of the probe and on the other hand that the gas flow in the sniffer unit is not hindered by calibration. When making measurements using the sniffer technique (see Section 5.7.2) it is also necessary to take into account the distance from the probe tip to the surface of the specimen and the scanning speed; these must be included as a part of the calibration. In the special case where helium concentration is being measured, calibration can be made using the helium content in the air, which is a uniform 5 ppm world-wide.

Test leaks (also known as standard leaks or reference leaks) normally comprise a gas supply, a choke with a defined conductance value, and a valve. The configuration will be in accordance with the test leak rate required. Figure 5.9 shows various test leaks. Permeation leaks are usually used for leak rates of $10^{-10} < Q_L < 10^{-7}$, capillaries, between 10^{-8} and 10^{-4} and, for very large leak rates in a range from 10 to 1000 mbar · l/s, pipe sections or orifice plates with exactly defined conductance values (dimensions).

Test leaks used with a refrigerant charge represent a special situation since the refrigerants are liquid at room temperature. Such test leaks have a supply space for liquid from which, through a shut-off valve, the space filled only with the refrigerant vapor (saturation vapor pressure) can be reached, ahead of the capillary leak. One technological problem which is difficult to solve is posed by the fact that all refrigerants are also very good solvents for oil and grease and thus are often seriously contaminated so that it is difficult to fill the test leaks with pure refrigerant. Decisive here is not only the chemical composition but above all dissolved particles which can repeatedly clog the fine capillaries.

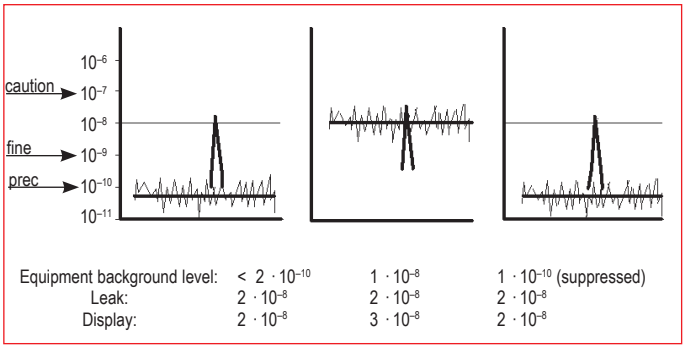


Fig. 5.8 Example of zero-point suppression



a Reference leak without gas supply, TL4, TL6
b Reference leak for sniffer and vacuum applications, TL4-6
c (Internal) capillary test leak TL7
d Permeation (diffusion) reference leak, TL8
e Refrigerant calibrated leak

Fig. 5.9 Examples for the construction of test leaks

5.5.2.4 Leak detectors with quadrupole mass spectrometer (ECOTEC II)

INFICON builds leak detectors with quadrupole mass spectrometers to register masses greater than helium. Apart from special cases, these will be refrigerants. These devices thus serve to examine the tightness of refrigeration units, particularly those for refrigerators and air conditioning equipment.

Figure 4.2 shows a functional diagram for a quadrupole mass spectrometer. Of the four rods in the separation system, the two pairs of opposing rods will have identical potential and excite the ions passing through along the center line so that they oscillate transversely. Only when the amplitude of these oscillations remains smaller than the distance between the rods can the appropriate ion pass through the system of rods and ultimately reach the ion trap, where it will discharge and thus be counted. The flow of electrons thus created in the line forms the measurement signal proper. The other ions come into contact with one of the rods and will be neutralized there.

Figure 5.10 shows the vacuum schematic for an ECOTEC II. The mass spectrometer (4) only operates under high vacuum conditions, i.e. the pressure here must always remain below 10^{-4} mbar. This vacuum is generated by the turbomolecular pump (3) with the support of the diaphragm pump (1). The pressure PV between the two pumps is measured with a piezo resistive measuring system (2) and this pressure lies in the range between 1 to 4 mbar while in the measurement mode. This pressure must not exceed a value of 10 mbar as otherwise the turbomolecular pump will not be capable of maintaining the vacuum in the mass spectrometer. The unit can easily be switched over at the control unit from helium to any of various refrigerants, some of which may be selected as desired. Naturally the unit must be calibrated separately for each of these masses. Once set, however, the values remain available in storage so that after calibration has been effected for all the gases (and a separate reference leak is required for each gas!) it is possible to switch directly from one gas to another.

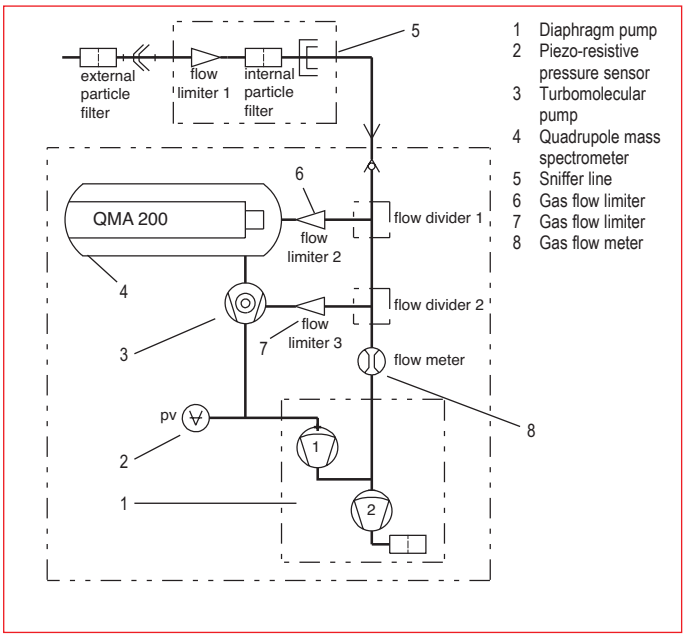


Fig. 5.10 Vacuum schematic for the ECOTEC II

5.5.2.5 Helium leak detectors with 180° sector mass spectrometer (UL 200, UL 500)

These units are the most sensitive and also provide the greatest degree of certainty. Here "certain" is intended to mean that there is no other method with which one can, with greater reliability and better stability, locate leaks and measure them quantitatively. For this reason helium leak detectors, even though the purchase price is relatively high, are often far more economical in the long run since much less time is required for the leak detection procedure itself.

A helium leak detector comprises basically two sub-systems in portable units and three in stationary units. These are:

1. the mass spectrometer
2. the high vacuum pump and
3. the auxiliary roughing pump system in stationary units.

The mass spectrometer (see Fig. 5.11) comprises the ion source (1–4) and the deflection system (5–9). The ion beam is extracted through the orifice plate (5) and enters the magnetic field (8) at a certain energy level. Inside the magnetic field the ions move along circular paths whereby the radius for a low mass is smaller than that for higher masses. With the correct setting of the acceleration voltage during **tuning** one can achieve a situation in which the ions describe a circular arc with a defined curvature radius. Where mass 4 (helium) is involved, they pass thorough the aperture (9) to the ion trap (13). In some devices the discharge current for the ions impinging upon the total pressure electrodes will be measured and evalu-

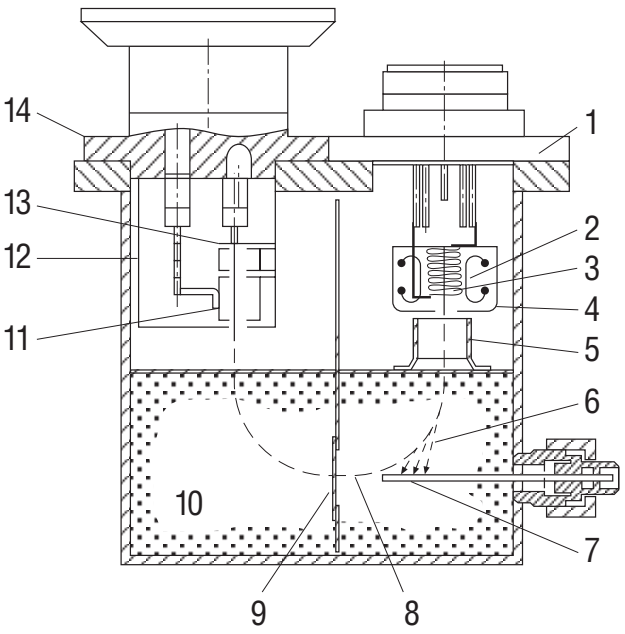


Fig. 5.11 Configuration of the 180° sector mass spectrometer

ated as a total pressure signal. Ions with masses which are too small or too great should not be allowed to reach the ion trap (13) at all, but some of these ions will do so in spite of this, either because they are deflected by collisions with neutral gas particles or because their initial energy deviates too far from the required energy level. These ions are then sorted out by the suppressor (11) so that only ions exhibiting a mass of 4 (helium) can reach the ion detector (13). The electron energy at the ion source is 80 eV. It is kept this low so that components with a specific mass of 4 and higher – such as multi-ionized carbon or quadruply ionized oxygen – cannot be created. The ion sources for the mass spectrometer are simple, rugged and easy to replace. They are heated continuously during operation and are thus sensitive to contamination. The two selectable yttrium oxide coated iridium cathodes have a long service life. These cathodes are largely insensitive to air ingress, i.e. the quick-acting safety cut-out will keep them from burning out even if air enters. However, prolonged use of the ion source may eventually lead to cathode embrittlement and can cause the cathode to splinter if exposed to vibrations or shock.

Depending on the way in which the inlet is connected to the mass spectrometer, one can differentiate between two types of MSLD.

5.5.2.6 Direct-flow and counter-flow leak detectors

Figure 5.12 shows the vacuum schematic for the two leak detector types. In both cases the mass spectrometer is evacuated by the high vacuum pumping system comprising a turbomolecular pump and a rotary vane pump. The diagram on the left shows a **direct-flow leak detector**. Gas from the inlet port is admitted to the spectrometer via a cold trap. It is actually equivalent to a cryopump in which all the vapors and other contaminants condense. (The cold trap in the past also provided effective protection against the oil vapors of the diffusion pumps used at that time). The auxiliary roughing pump system serves to pre-evacuate the components to be tested or the connector line between the leak detector and the system to be tested. Once the relatively low inlet pressure (pumping time!) has been reached, the valve between the auxiliary pumping system and the cold trap will be opened for the measurement. The Seff used in equation 5.4b is the pumping speed of the turbomolecular pump at the ion source location:

$$Q_{He} = p_{He} \cdot S_{eff,\text{turbomolecular pump ion source}} \qquad (5.5a)$$

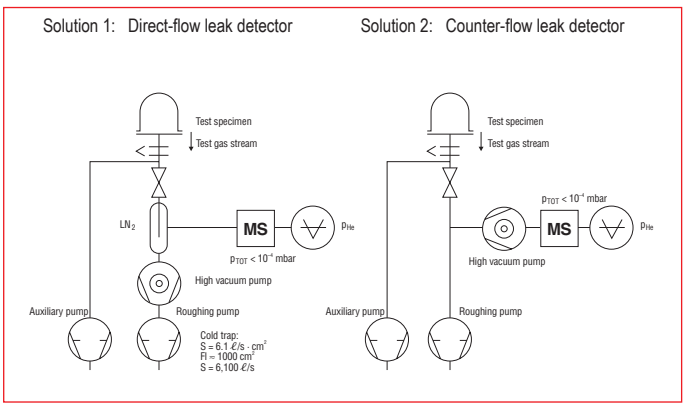


Fig. 5.12 Full-flow and counter-flow leak detector

In the case of direct-flow leak detectors, an increase in the sensitivity can be achieved by reducing the pumping speed, for example by installing a throttle between the turbomolecular pump and the cold trap. This is also employed to achieve maximum sensitivity. To take an example:

The smallest detectable partial pressure for helium is $p_{min,He} = 1 \cdot 10^{-12}$ mbar. The pumping speed for helium would be $S_{He} = 10$ l/s. Then the smallest detectable leak rate is $Q_{min} = 1 \cdot 10^{-12}$ mbar $\cdot$ 10 l/s = $1 \cdot 10^{-11}$ mbar $\cdot$ l/s. If the pumping speed is now reduced to 1/s, then one will achieve the smallest detectable leak rate of $1 \cdot 10^{-12}$ mbar $\cdot$ l/s. One must keep in mind, however, that with the increase in the sensitivity the time constant for achieving a stable test gas pressure in the test specimen will be correspondingly larger (see Section 5.5.2.9).

In Figure 5.12 the right hand diagram shows the schematic for the **counter-flow leak detector**. The mass spectrometer, the high vacuum system and also the auxiliary roughing pump system correspond exactly to the configuration for the direct-flow arrangement. The feed of the gas to be examined is however connected between the roughing pump and the turbomolecular pump. Helium which reaches this branch point after the valve is opened will cause an increase in the helium pressure in the turbomolecular pump and in the mass spectrometer. The pumping speed S_{eff} inserted in equation 5.4b is the pumping speed for the rotary vane pump at the branch point. The partial helium pressure established there, reduced by the helium compression factor for the turbomolecular pump, is measured at the mass spectrometer. The speed of the turbomolecular pump in the counter-flow leak detectors is regulated so that pump compression also remains constant. Equation 5.5b is derived from equation 5.5a:

$$Q_{He} = p_{He} \cdot S_{eff} \cdot K \qquad (5.5b)$$

S_{eff} = effective pumping speed at the rotary vane pump at the branching point

K = Helium compression factor at the turbomolecular pump

The counter-flow leak detector is a particular benefit for automatic vacuum units since there is a clearly measurable pressure at which the valve can be opened, namely the roughing vacuum pressure at the turbomolecular pump. Since the turbomolecular pump has a very large compression capacity for high masses, heavy molecules in comparison to the light test gas, helium (M = 4), can in practice not reach the mass spectrometer. The turbomolecular pump thus provides ideal protection for the mass spectrometer and thus eliminates the need for an LN_2 cold tap, which is certainly the greatest advantage for the user. Historically, counter-flow leak detectors were developed later. This was due in part to inadequate pumping speed stability, which for a long time was not sufficient with the rotary vane pumps used here. For both types of leak detector, stationary units use a built-in auxiliary pump to assist in the evacuation of the test port. With portable leak detectors, it may be necessary to provide a separate, external pump, this being for weight reasons.

5.5.2.7 Partial flow operation

Where the size of the vacuum vessel or the leak makes it impossible to evacuate the test specimen to the necessary inlet pressure, or where this would simply take too long, then supplementary pumps will have to be used. In this case the helium leak detector is operated in accordance with

the so-called “partial flow” concept. This means that usually the larger part of the gas extracted from the test object will be removed by an additional, suitably dimensioned pump system, so that only a part of the gas stream reaches the helium leak detector (see Fig. 5.13). The splitting of the gas flow is effected in accordance with the pumping speed prevailing at the branching point. The following then applies:

$$Q_{\text{Vacuum vessel}} = \gamma \cdot \text{Display}_{\text{Leak detector}} \qquad (5.6)$$

where g is characterized as the partial flow ratio, i.e. that fraction of the overall leak current which is displayed at the detector. Where the partial flow ratio is unknown, g can be determined with a reference leak attached at the vacuum vessel:

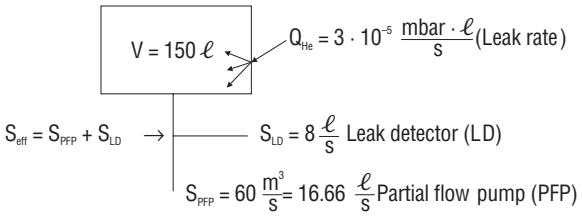
$$\gamma = \frac{\text{Display at the leak detector}}{Q_L \text{ for the reference leak}} \qquad (5.7)$$

5.5.2.8 Connection to vacuum systems

The partial flow concept is usually used in making the connection of a helium leak detector to vacuum systems with multi-stage vacuum pump sets. When considering where to best make the connection, it must be kept in mind that these are usually small, portable units which have only a low pumping speed at the connection flange (often less than 1 l/s). This makes it all the more important to estimate – based on the partial flow ratio to be expected vis à vis a diffusion pump with pumping speed of 12000 l/s, for example – which leak rates can be detected at all. In systems with high vacuum- and Roots pumps, the surest option is to connect the leak detector between the rotary vane pump and the roots pump or between the roots pump and the high vacuum pump. If the pressure there is greater than the permissible inlet pressure for the leak detector, then the leak detector will have to be connected by way of a metering (variable leak) valve. Naturally one will have to have a suitable connector flange available. It is also advisable to install a valve at this point from the outset so that, when needed, the leak detector can quickly be coupled (with the system running) and leak detection can commence immediately after opening the valve. In order to avoid this valve being opened inadvertently, it should be sealed off with a blank flange during normal vacuum system operation.

A second method for coupling to larger systems, for example, those used for removing the air from the turbines in power generating stations, is to couple at the discharge. A sniffer unit is inserted in the system where it discharges to atmosphere. One then sniffs the increase in the helium concentration in the exhaust. Without a tight coupling to the exhaust, however, the detection limit for this application will be limited to 5 ppm, the natural helium content in the air. In power plants it is sufficient to insert the tip of the probe at an angle of about 45 ° from the top into the discharge line (usually pointing upward) of the (water ring) pump.

Partial flow principle (example)



A) Signal amplitude:
Splitting of the gas flow (also of the test gas!) in accordance with the effective pumping speed at the partial flow branch point

Overal pumping speed: $S_{eff} = S_{LD} + S_{PFP} = 8 + 16.66 = 24.66$ ℓ/s

γ ... Partial flow ratio

Signal to Leak detector: $3 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}} \cdot \frac{8 \frac{\ell}{\text{s}}}{(8 + 16.66) \frac{\ell}{\text{s}}} = 9.73 \cdot 10^{-6} \frac{\text{mbar} \cdot \ell}{\text{s}}$

Signal to partial flow pump: $3 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}} \cdot \frac{16.66 \frac{\ell}{\text{s}}}{(8 + 16.66) \frac{\ell}{\text{s}}} = 2.02 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}}$

Check: Overall signal $Q_{He} = Q_{LD} + Q_{PFP}$ $= 3.00 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}}$

Partial flow ratio = Fraction of the overall flow to the leak detector

$$\gamma = \frac{Q_{LD}}{Q_{He}} = \frac{Q_{LD}}{Q_{LD} + Q_{PFP}} = \frac{1}{1 + \frac{Q_{PFP}}{Q_{LD}}}$$

or $$\gamma = \frac{S_{LD}}{S_{LD} + S_{PFP}} = \frac{1}{1 + \frac{S_{PFP}}{S_{LD}}}$$

$Q_{LD} = \gamma \cdot Q_{He}$ (Display, Leak rate)

B) Response time: $t_{95\%} = 3 \cdot \frac{V}{S_{eff}} = 3 \cdot \frac{150\ \ell}{24.66 \frac{\ell}{\text{s}}} = 18.25$ s

Estimate: Value for S, V and γ are uncertain → certain: calibrate with reference leak

Fig. 5.13 Partial flow principle

5.5.2.9 Time constants

The time constant for a vacuum system is set by

$$\tau = \frac{V}{S_{eff}} \qquad (5.8)$$

τ = Time constant
V = Volume of the container
S_{eff} = Effective pumping speed, at the test object

Figure 5.14 shows the course of the signal after spraying a leak in a test specimen attached to a leak detector, for three different configurations:

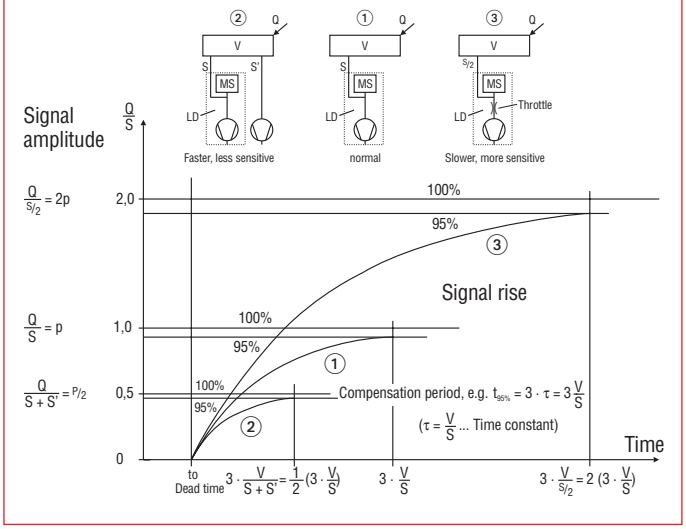


Fig. 5.14 Signal responses and pumping speed

1. Center: The specimen with volume of V is joined directly with the leak detector LD (effective pumping speed of S).
2. Left: In addition to 1, a partial flow pump with the same effective pumping speed, $S^{l} = S$, is attached to the test specimen.
3. Right: As at 1, but S is throttled down to 0.5◊S.

The signals can be interpreted as follow:

1: Following a “dead period” (or “delay time”) up to a discernible signal level, the signal, which is proportional to the partial pressure for helium, will rise to its full value of $p_{He} = Q/S_{eff}$ in accordance with equation 5.9:

$$p_{He} = \frac{Q}{S_{eff}} \cdot \left(1 - e^{\frac{-t}{\tau}}\right) \qquad (5.9)$$

The signal will attain a prortion of its ultimate value after

t = 1 τ . . 63.3 %	t = 2 τ . . 86.5 %
t = 3 τ . . 95.0 %	t = 4 τ . . 98.2 %
t = 5 τ . . 99.3 %	t = 6 τ . . 99.8 %

The period required to reach 95 % of the ultimate value is normally referred to as the **response time**.

2: With the installation of the partial flow pump both the time constant and the signal amplitude will be reduced by a factor of 2; that means a quicker rise but a signal which is only half as great. A small time constant means quick changes and thus quick display and, in turn, short leak detection times.

3: The throttling of the pumping speed to 0.5 S, increases both the time constant and the signal amplitude by a factor of 2. A large value for t thus increases the time required appropriately. Great sensitivity, achieved by reducing the pumping speed, is always associated with greater time requirements and thus by no means is always of advantage.

An estimate of the overall time constants for several volumes connected one behind to another and to the associated pumps can be made in an initial approximation by adding the individual time constants.

5.6 Limit values / Specifications for the leak detector

1. The **smallest detectable leak rate**.
2. The **effective pumping speed at the test connection**.
3. The **maximum permissible pressure inside the test specimen** (also the maximum permissible inlet pressure). This pressure p_{max} will be about 10^{-1} for LDs with classical PFPs and about 2 to 10 mbar for LDs with compound PFPs. The product of this maximum permissible operating pressure and the pumping speed S of the pump system at the detector’s test connection is the **maximum permissible throughput**:

$$Q_{max} = p_{max} \cdot S_{eff,\ connector} \qquad (5.10)$$

This equation shows that it is by no means advantageous to attain high sensitivity by throttling down the pumping speed. The maximum permissible throughput would otherwise be too small. The unit is not functional when – either due to one large leak or several smaller leaks – more gas flows into the unit than the maximum permissible throughput rate for the leak detector.

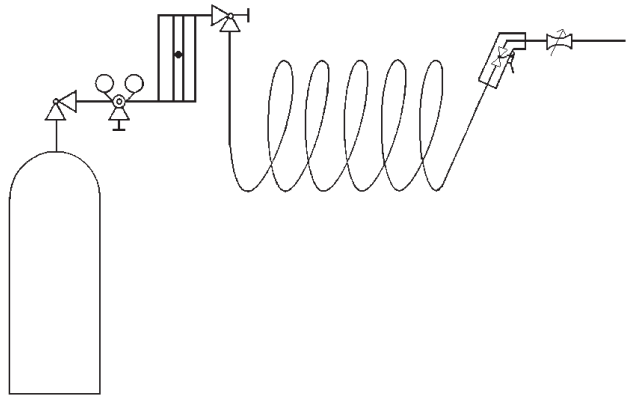
5.7 Leak detection techniques using helium leak detectors

5.7.1 Spray technique (local leak test)

The test specimen, connected to the helium leak detector, is slowly traced with a very fine stream of helium from the spray pistol, aimed at likely leakage points (welding seams, flange connectors, fused joints), bearing in mind the time constant of the system as per Equation 5.8 (see Fig. 5.14). The volume sprayed must be adjusted to suit the leak rate to be detected and the size and accessibility of the object being tested. Although helium is lighter than air and therefore will collect beneath the ceiling of the room, it will be so well distributed by drafts and turbulence induced by movements within the room that one need not assume that helium will be found primarily (or only) at the top of the room during search for leaks. In spite of this, it is advisable, particularly when dealing with larger components, to start the search for leaks at the top.

In order to avoid a surge of helium when the spray valve is opened (as this would “contaminate” the entire environment) it is advisable to install a choke valve to adjust the helium quantity, directly before or after the spray pistol (see Fig. 5.15). The correct quantity can be determined easiest by submerging the outlet opening in a container of water and setting the valve on the basis of the rising bubbles. Variable-area flowmeters are indeed available for the required small flow quantities but are actually too expensive. In addition, it is easy to use the water-filled container at any time to determine whether helium is still flowing.

The helium content of the air can also be detected with helium leak detectors where large leaks allow so much air to enter the vessel that the



Avoiding the “helium surge” when the pistol valve is opened
a) Throttle hose or
b) Adjustable throttle valve ahead of the spay pistol
Minimum helium quantity for correct display: Changing the setting for the throttle shall not affect indication.
The minimum quantity is always much smaller than one would set without a flowmeter (e.g. by listening for flow or letting the helium flow across moistened lips). The simplest check without a flowmeter: Letting gas bubble through water.

Fig. 5.15 Helium spray equipment

5 ppm share of helium in the air is sufficient for detection purposes. The leak rate is then:

$$\frac{\text{Display (pure He)}}{1} = \frac{\text{Display (atmosph. He)}}{5 \cdot 10^{-6}}$$

$$Q_L = \text{Display (pure He)} \qquad (5.11)$$
$$= 2 \cdot 10^{+5} \cdot \text{Display (atmospheric He)}$$

5.7.2 Sniffer technology (local leak test using the positive pressure method)

Here the points suspected of leaking at the pressurized test specimen (see Fig. 5.4, d) are carefully traced with a test gas probe which is connected with the leak detector by way of a hose. Either helium or hydrogen can be detected with the INFICON helium leak detectors. The sensitivity of the method and the accuracy of locating leaky points will depend on the nature of the sniffer used and the response time for the leak detector to which it is connected. In addition, it will depend on the speed at which the probe is passed by the leak points and the distance between the tip of the probe and the surface of the test specimen. The many parameters which play a part here make it more difficult to determine the leak rates quantitatively. Using sniffer processes it is possible, virtually independent of the type of gas, to detect leak rates of about 10^{-7} mbar · l/s. The limitation of sensitivity in the detection of helium is due primarily to the helium in the atmosphere (see Chapter 9, Table VIII). In regard to quantitative measurements, the leak detector and sniffer unit will have to be calibrated together. Here the distance from the specimen and the tracing speed will have to be included in calibration, too.

5.7.3 Vacuum envelope test (integral leak test)

Vacuum envelope tests are integral leak tests using helium as the test gas, in which the test specimen is enclosed either in a rigid (usually metal) enclosure or in a light plastic envelope. The helium which enters or leaves (depending on the nature of the test) the test specimen is passed to a helium leak detector, where it is measured. Envelope tests are made either with the test specimen pressurized with helium (Fig. 5.4c) or with the test specimen evacuated (Fig. 5.4a). In both cases it may be necessary to convert the helium enrichment figure (accumulation) to the helium standard leak rate.

5.7.3.1 Envelope test – test specimen pressurized with helium

a) Envelope test with concentration measurement and subsequent leak rate calculation

To determine overall leakiness of a test object pressurized with helium the object shall be enclosed in an envelope which is either rigid or deformable (plastic). The test gas leaving the leaks accumulates so that the helium concentration in the envelope rises. Following an enrichment period to be determined (operating period) the change in concentration inside the envelope will be measured with a sniffer connected to the helium detection unit. The overall leak rate (integral leak rate) can be calculated following calibration of the test configuration with a reference concentration, e.g. atmospheric air. This method makes it possible to detect even the smallest overall leakiness and is suitable in particular for automated industrial leak testing. Due to gas accumulation, the limits for normal sniffer techniques are shifted toward lower leak rates and the ambient conditions such as temperature, air flow and sniffer tracing speed lose influence. When using plastic envelopes it is necessary to take into account helium permeation through the plastic envelope during long enrichment periods.

b) Direct measurement of the leak rate with the leak detector (rigid envelope)

When the test specimen, pressurized with helium, is placed in a rigid vacuum chamber, connected to a helium leak detector, the integral leak rate can be read directly at the leak detector.

5.7.3.2 Envelope test with test specimen evacuated

a) Envelope = “plastic tent”

The evacuated test specimen is surrounded by a light-weight (plastic) enclosure and this is then filled with helium once the atmospheric air has been removed. When using a plastic bag as the envelope, the bag should be pressed against the test specimen before filling it with helium in order to expel as much air as possible and to make the measurement with the purest helium charge possible. The entire outside surface of the test object is in contact with the test gas. If test gas passes through leaks and into the test specimen, then the integral leak rate will be indicated, regardless of the number of leaks. In addition, it is necessary to observe when repeating testing in enclosed areas that the helium content of the room will rise quite rapidly when the envelope is removed. Using plastic bags is thus more advisable for “one-off” testing of large plants. The plastic envelope used here is often referred to as a “tent”.

b) Rigid envelope

The use of a solid vacuum vessel as the rigid envelope, on the other hand, is better for repetitive testing where an integral test is to be made. When solid envelopes are used it is also possible to recover the helium once the test has been completed.

5.7.4 “Bombing” test, “Storage under pressure”

The “bombing” test is use to check the tightness of components which are already hermetically sealed and which exhibit a gas-filled, internal cavity. The components to be examined (e.g. transistors, IC housings, dry-reed relays, reed contact switches, quartz oscillators, laser diodes and the like) are placed in a pressure vessel which is filled with helium. Operating with the test gas at relatively high pressure (5 to 10 bar) and leaving the system standing over several hours the test gas (helium) will collect inside the

leaking specimens. This procedure is the actual “bombing”. To make the leak test, then, the specimens are placed in a vacuum chamber following “bombing”, in the same way as described for the vacuum envelope test. The overall leak rate is then determined. Specimens with large leaks will, however, lose their test gas concentration even as the vacuum chamber is being evacuated, so that they will not be recognized as leaky during the actual leak test using the detector. It is for this reason that another test to register very large leaks will have to be made prior to the leak test in the vacuum chamber.

5.8 Industrial leak testing

Industrial leak testing using helium as the test gas is characterized above all by the fact that the leak detection equipment is fully integrated into the manufacturing line. The design and construction of such test units will naturally take into account the task to be carried out in each case (e.g. leak testing vehicle rims made of aluminum or leak testing for metal drums). Mass-produced, standardized component modules will be used wherever possible. The parts to be examined are fed to the leak testing system (envelope test with rigid envelope and positive pressure [5.7.3.1b] or vacuum [5.7.3.2b] inside the specimen) by way of a conveyor system. There they will be examined individually using the integral methods and automatically moved on. Specimens found to be leaking will be shunted to the side.

The advantages of the helium test method, seen from the industrial point of view, may be summarized as follows:

- The leak rates which can be detected with this process go far beyond all practical requirements.
- The integral leak test, i.e. the total leak rate for all individual leaks, facilitates the detection of microscopic and sponge-like distributed leaks which altogether result in leakage losses similar to those for a larger individual leak.
- The testing procedure and sequence can be fully automated.
- The cyclical, automatic test system check (self-monitoring) of the device ensures great testing reliability.
- Helium is non-toxic and non-hazardous (no maximum allowable concentrations need be observed).
- Testing can be easily documented, indicating the parameters and results, on a printer.

Use of the helium test method will result in considerable increases in efficiency (cycling times being only a matter of seconds in length) and lead to a considerable increase in testing reliability. As a result of this and due to the EN/ISO 9000 requirements, traditional industrial test methods (water bath, soap bubble test, etc.) will now largely be abandoned.

6 Thin film controllers and control units with quartz oscillators

6.1 Introduction

It took a long time to go from the coating of quartz crystals for frequency fine tuning, which has long been in practice, to utilization of frequency change to determine the mass per unit area as a microbalance with the present-day degree of precision. In 1880 two brothers, J. and P. Curie, discovered the piezoelectric effect. Under mechanical loads on certain quartz crystal surfaces, electrical charges occur that are caused by the asymmetrical crystalline structure of SiO_2. Conversely, in a piezocrystal deformations appear in an electrical field and mechanical oscillations occur in an alternating field. A distinction is made between bending oscillations, thickness shear mode and thickness shear oscillations. Depending on the orientation of the cut plane to the crystal lattice, a number of different cuts are distinguished, of which only the so-called AT cut with a cut angle of 35°10“ is used in thin film controllers because the frequency has a very low temperature dependence in the range between 0 and 50 °C with this cut. Accordingly, an attempt must be made not to exceed this temperature range during coating (water cooling of crystal holder).

Since there is still a problem with “quartz capacity” (i.e. the maximum possible coating thickness of the quartz at which it still oscillates reliably) despite refined technology, a number of approaches have been developed to expand this capacity:

1. The use of several crystals, one behind the other, in a multiple crystal holder with automatic change and data updating in the event of imminent failure of a quartz: **CrystalSix**.
2. The **RateWatcher** function, in which the quartz is alternately exposed to the coating beam for a short time until all measurements and regulation have been carried out and then remains covered by a shutter for a longer period of time.

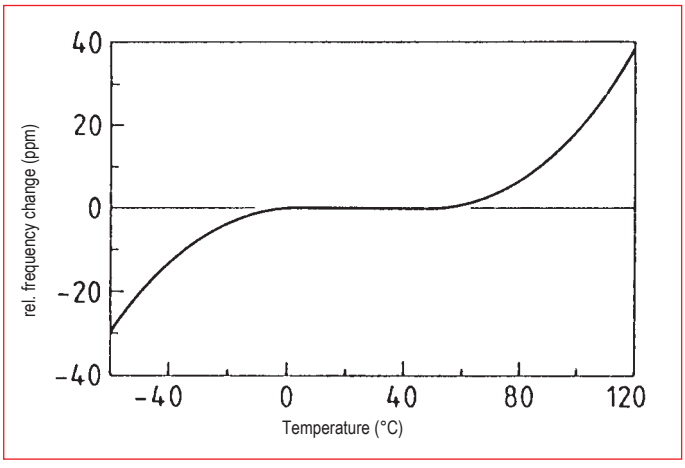


Fig. 6.1 Natural frequency as a function of temperature in an AT cut quartz crystal

The selection of the “right” crystal holder thus plays an important role in all measurements with quartz oscillators. Various crystal holder designs are recommended for the different applications: with or without shutter, bakeable for UHV, double crystal holder or crystal six as well as special versions for sputter applications. In addition to these important and more “mechanical” aspects, the advances in measuring and control technology and equipment features will be discussed in the following.

6.2 Basic principles of coating thickness measurement with quartz oscillators

The quartz oscillator coating thickness gauge (thin film controller) utilizes the piezoelectric sensitivity of a quartz oscillator (monitor crystal) to the supplied mass. This property is utilized to monitor the coating rate and final thickness during vacuum coating.

A very sharp electromechanical resonance occurs at certain discrete frequencies of the voltage applied. If mass is added to the surface of the quartz crystal oscillating in resonance, this resonance frequency is diminished. This frequency shift is very reproducible and is now understood precisely for various oscillation modes of quartz. Today this phenomenon, which is easy to understand in heuristic terms, is an indispensable measuring and process control tool, with which a coating increase of less than one atomic layer can be detected.

In the late 1950s Sauerbrey and Lostis discovered that the frequency shift connected with the coating of the quartz crystal is a function of the change in mass due to the coating material in the following way:

$$\frac{M_f}{M_q} = \frac{\Delta F}{F_q} \quad \text{or} \quad M_f = M_q \cdot \frac{\Delta F}{F_q} \quad \text{with} \qquad (6.1)$$

M_f mass of the coating
M_q mass of the quartz prior to coating
F_q frequency prior to coating
F_c frequency after coating
$\Delta F = F_f - F_c$ ... frequency shift due to coating

If the following are now applied: $M_f = (M_c - M_q) = D_f \cdot \rho_f \cdot A$ and $M_q = Dq \cdot \rho_q \cdot A$, where T = the coating thickness, ρ = density and A stands for area while the index q stands for the state of the “uncoated quartz” and c for the state after “frequency shift due to coating”, the following results are obtained for the coating thickness:

$$D_f = \frac{F_q}{F_q} \cdot D_q \cdot \rho_q \cdot \frac{\Delta F}{F_q \cdot \rho_f} = K \cdot \frac{\Delta F}{\rho_f} \quad \text{with}$$

$$K = \frac{D_q \cdot F_q \cdot \rho_q}{F_q^2} = \frac{N_{AT} \cdot \rho_q}{F_q^2} \quad \text{where}$$

$N = F_q \cdot D_q$ is the frequency constant (for the AT cut N_{AT} = 166100 Hz · cm) and ρ_q = 2.649 g/cm³ is the density of the quartz. The coating thickness is thus proportional to the frequency shift ΔF and inversely proportional to the

Thin film controllers/control units

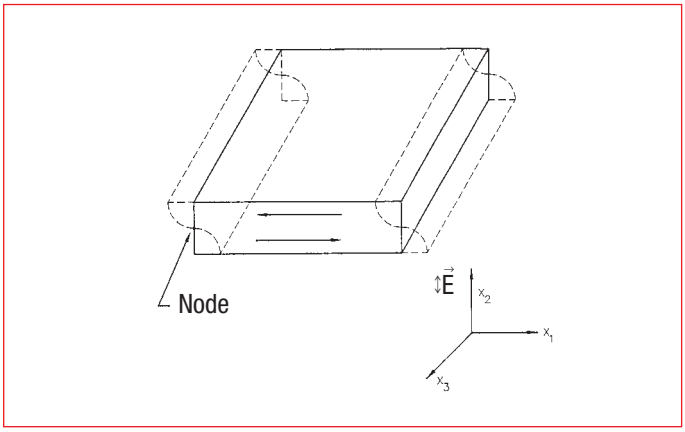


Fig. 6.2 Thickness shear oscillations

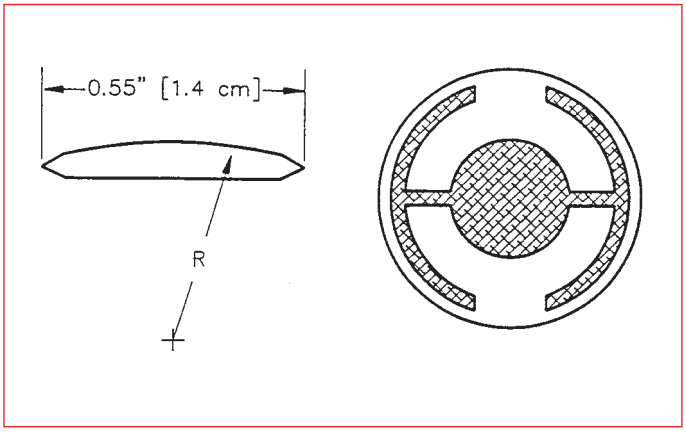


Fig. 6.3 Shape of LEYBOLD-Inficon quartz crystals

density ρ_f of the coating material. The equation

$$D_f = K \cdot \frac{\Delta F}{\rho_f}$$

for the coating thickness was used in the first coating thickness measuring units with "frequency measurement" ever used. According to this equation, a crystal with a starting frequency of 6.0 MHz displays a decline in frequency of 2.27 Hz after coating with 1Å of aluminum (de = 2.77 g/cm^3). In this way the growth of a fixed coating due to evaporation or sputtering can be monitored through precise measurement of the frequency shift of the crystal. It was only when knowledge of the quantitative interrelationship of this effect was acquired that it became possible to determine precisely the quantity of material that is deposited on a substrate in a vacuum. Previously this had been practically impossible.

6.3 The shape of quartz oscillator crystals

Regardless of how sophisticated the electronic environment is, the main component for coating measurement remains the monitor quartz crystal. Originally monitor quartzes had a square shape. Fig. 6.4 shows the resonance spectrum of a quartz resonator with the design used today (Fig. 6.3). The lowest resonance frequency is initially given by a thickness shear oscillation, which is called the fundamental wave. The characteristic

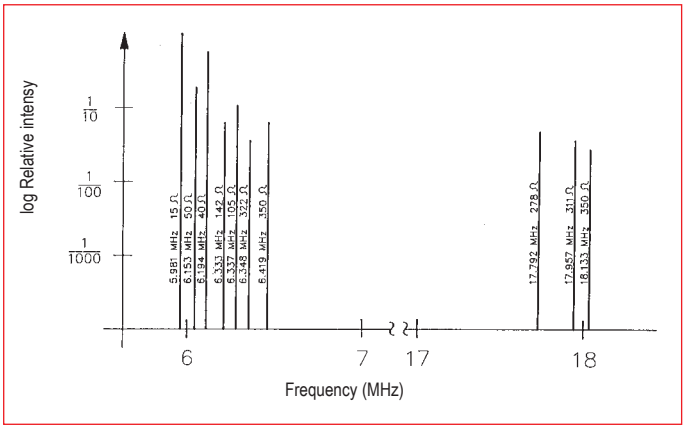


Fig. 6.4 Frequency resonance spectrum

motions of the thickness shear oscillation are parallel to the main crystal boundary surfaces. In other words: the surfaces are shift antinodes, see Fig. 6.2. The resonance frequencies slightly above the basic frequency are called "anharmonic" and are a combination of thickness shear and thickness rotation oscillation forms. The resonance frequency at around three times the value for the fundamental wave is called "quasi-harmonic". Near the quasi-harmonic there are also a number of anharmonics with a slightly higher frequency.

The design of the monitor crystals used nowadays (see Fig. 6.3) displays a number of significant improvements over the original square crystals. The first improvement was the use of round crystals. The enlarged symmetry greatly reduced the number of possible oscillation modes. A second group of improvements involved providing one of the surfaces with a contour and making the excitation electrode smaller. The two together ensure that the acoustic energy is recorded. Reducing the electrode diameter limits the excitation to the middle area. The surface contour consumes the energy of the moving acoustic waves before they reach the crystal edge. It is not reflected into the center where it could interfere with new incoming waves.

Such a small crystal behaves like an infinitely expanded crystal. However, if the crystal vibrations remain restricted to the center, one can clamp the outer edge to a crystal holder, without engendering undesired side effects. Moreover, contouring reduces the resonance intensity of undesired anharmonics. This limits the capacity of the resonator to maintain these oscillations considerably.

Use of an adhesive coating has enhanced the adhesion of the quartz electrode. Even the rate spikes occurring with increasing film stress (strain) and caused by micro-tears in the coating were reduced. Coating material remains at these micro-tears without adhesion and therefore cannot oscillate. These open areas are not registered and thus an incorrect thickness is indicated.

Fig. 6.4 shows the frequency behavior of a quartz crystal shaped as in Fig. 6.3. The ordinate represents the amplitude of the oscillation or also the current flowing through the crystal as a function of the frequency on the abscissa.

Usually an AT cut is chosen for the coating thickness measurement because through the selection of the cut angle the frequency has a very small temperature coefficient at room temperature.

Since one cannot distinguish between

- coating: frequency reduction = negative influence
- temperature change:
  negative or positive influence
- temperature gradients on the crystal, positive or negative
- stresses caused by the coating

it is important to minimize the temperature influence. This is the only way to measure small differences in mass.

6.4 Period measurement

Although the instruments that functioned according to equation 6.2 were very useful, it soon became obvious that for the desired accuracy their area of application was typically limited to $\Delta F < 0.02\ F_q$. Even at a relative frequency change of $(F_q - F_c) / F_q < 2$ %, errors of around 2 % occurred in the coating thickness measurement so that the „usable service life“ of the coating in the case of a 6-MHz monitor crystal was about 120 kHz.

In 1961 Behrndt discovered that:

$$\frac{M_f}{M_q} = \frac{(T_c - T_q)}{T_q} = \frac{\Delta F}{F_c} \qquad \text{with (6.3)}$$

$T_c = 1 / F_c$ ... oscillation period, coated
$T_q = 1 / F_q$ ... oscillation period, uncoated

The period measurement (measurement of the oscillation duration) was the result of the introduction of digital time measurement and the discovery of the proportionality of crystal thickness D_q and oscillation duration T_q. The necessary precision of thickness measurement permits application of equation 6.3 up to about $\Delta F < 0.05\ F_q$.

In period measurement a second crystal oscillator is essentially used as a reference oscillator that is not coated and usually oscillates at a much higher frequency than the monitor crystal. The reference oscillator generates small precision time intervals, with which the oscillation duration of the monitor crystal is determined. This is done by means of two pulse counters: the first counts a fixed number of monitor oscillations m. The second is started simultaneously with the first and counts the oscillations of the reference crystal during m oscillations of the monitor crystal. Because the reference frequency F_r is known and stable, the time for m monitor oscillations can be determined accurately to $\pm 2/F_r$. The monitor oscillation period is then

$$\frac{n}{F_r \cdot m}$$

where n is the reading of the reference counter. The accuracy of the measurement is determined by the frequency of the reference oscillator and the length of the counting time that is specified through the size of m.

For low coating rates, small densities of the coating material and fast measurements (that require short counting times), it is important to have a reference oscillator with a high frequency. All of this requires great time precision so that the small coating-related frequency shifts can be resolved. If the frequency shift of the monitor crystal decreases between two measurements on the order of magnitude of the frequency measurement accuracy, good rate regulation becomes impossible (rate regulation: regulation of the energy supply to the coating source so that a specified coating thickness growth per time unit is maintained). The great measurement uncertainty then causes more noise in the closed loop, which can only be countered with longer time constants. This in turn makes the corrections due to system deviation slow so that relatively long deviations from the desired rate result. This may not be important for simple coatings, but for critical coatings, as in the case of optical filters or very thin, slowly growing single-crystal coatings, errors may result. In many cases, the desired properties of such coatings are lost if the rate deviations are more than one or two percent. Finally, frequency and stability of the reference oscillator determine the precision of the measurement.

6.5 The Z match technique

Miller and Bolef (1968) treated the quartz oscillator and coating system as a single-dimensional, coherent acoustic resonator. Lu and Lewis (1972) developed the simplified Z match equation on that basis. Simultaneous advances in electronics, particularly the microprocessor, made it possible to solve the Z match equation in real time. Most coating process control units sold today use this sophisticated equation, which takes into account the acoustic properties of the quartz oscillator/coating system:

$$T_f = \left(\frac{N_{AT} \cdot d_q}{\pi \cdot d_f \cdot F_c \cdot Z}\right) \cdot \operatorname{arctg}\left(Z \cdot \operatorname{tg}\left[\frac{\pi \cdot (F_q - F_c)}{F_q}\right]\right) \qquad (6.4)$$

$$Z = \frac{\sqrt{d_q \cdot U_q}}{d_f \cdot U_f} \qquad \text{acoustic impedance ratio}$$

U_q = shear module, quartz
U_f = shear module, film

This led to basic understanding of the conversion of frequency shift into thickness which enabled correct results in a practical time frame for process control. To achieve this high degree of accuracy, the user must only enter an additional material parameter Z_f for the coating material. The validity of the equation was confirmed for many materials and it applies to frequency shifts up to $\Delta F < 0.4\ F_q$! Note that equation 6.2 was only valid up to $\Delta F < 0.02\ F_q$. And equation 6.3 only up to $\Delta F < 0.05\ F_q$.

6.6 The active oscillator

All units developed up to now are based on use of an active oscillator, as shown schematically in Fig. 6.5. This circuit keeps the crystal actively in resonance so that any type of oscillation duration or frequency measurement can be carried out. In this type of circuit the oscillation is maintained as long as sufficient energy is provided by the amplifier to compensate for losses in the crystal oscillation circuit and the crystal can effect the necessary phase shift. The basic stability of the crystal oscillator is created through the sudden phase change that takes place near the series resonance point even with a small change in crystal frequency, see Fig. 6.6.

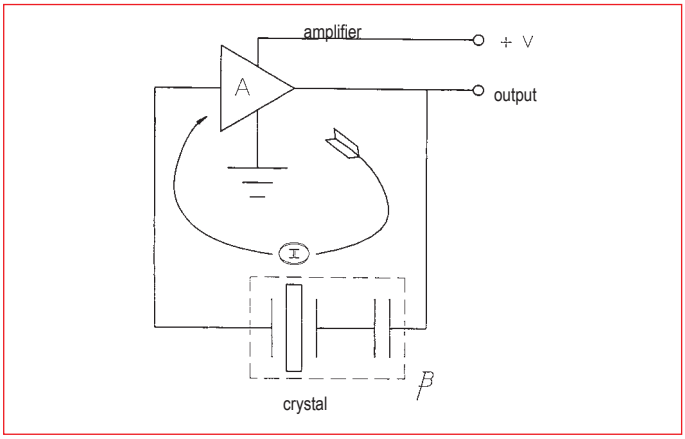


Fig. 6.5 Circuit of the active oscillator

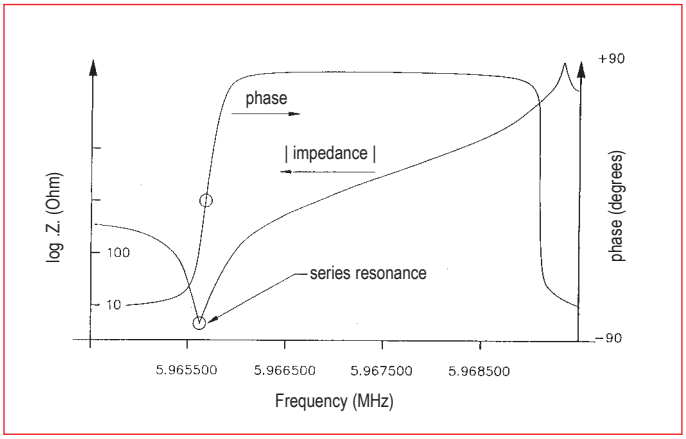


Fig. 6.6 Crystal frequencies near the series resonance point

Normally an oscillator circuit is designed such that the crystal requires a phase shift of 0 degrees to permit work at the series resonance point. Long- and short-term frequency stability are properties of crystal oscillators because very small frequency differences are needed to maintain the phase shift necessary for the oscillation. The frequency stability is ensured through the quartz crystal, even if there are long-term shifts in the electrical values that are caused by “phase jitter” due to temperature, ageing or short-term noise. If mass is added to the crystal, its electrical properties change.

Fig. 6.7 shows the same graph as Fig 6.6, but for a thickly coated crystal. It has lost the steep slope displayed in Fig. 6.6. Because the phase rise is less steep, any noise in the oscillator circuit leads to a larger frequency shift than would be the case with a new crystal. In extreme cases, the original phase/frequency curve shape is not retained; the crystal is not able to carry out a full 90 ° phase shift.

The impedance |Z| can increase to very high values. If this happens, the oscillator prefers to oscillate in resonance with an anharmonic frequency. Sometimes this condition is met for only a short time and the oscillator oscillation jumps back and forth between a basic and an anharmonic oscillation or it remains as an anharmonic oscillation. This phenomenon is well known as „mode hopping“. In addition to the noise of the rate signal created, this may also lead to incorrect termination of a coating because of the phase jump. It is important here that, nevertheless, the controller frequently continues to work under these conditions. Whether this has occurred can only be ascertained by noting that the coating thickness is suddenly significantly smaller, i.e. by the amount of the frequency difference between the fundamental wave and the anharmonic adopted by the oscillation.

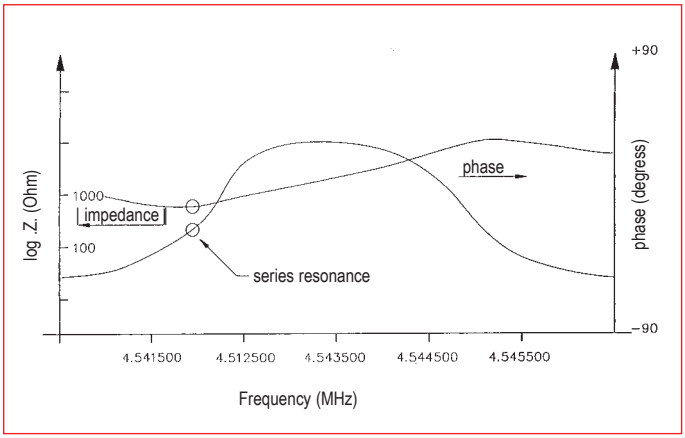


Fig. 6.7 Oscillations of a thickly coated crystal

6.7 The mode-lock oscillator

INFICON has developed a new technology for overcoming these constraints on the active oscillator. The new system constantly analyzes the response of the crystal to an applied frequency: not only to determine the (series) resonance frequency, but also to ensure that the quartz oscillates in the desired mode. The new system is insensitive to mode hopping and the resultant inaccuracy. It is fast and precise. The crystal frequency is determined 10 times a second with an accuracy to less than 0.0005 Hz.

The ability of the system to initially identify and then measure a certain mode opens up new opportunities thanks to the advantages of the additional information content of these modes. This new, „intelligent“ measuring device makes use of the phase/frequency properties of the quartz crystal to determine the resonance frequency. It works by applying a synthesized sinus wave of a certain frequency to the crystal and measuring the phase difference between the applied signal voltage and the current flowing through the crystal. In the case of series resonance, this difference is exactly zero degrees; then the crystal behaves like an ohmic resistance. By disconnecting the applied voltage and the current that returns from the crystal, one can determine with a phase comparator whether the applied frequency is higher or lower than the crystal resonance point.

The crystal impedance is capacitive at frequencies below the fundamental wave and inductive at frequencies above the resonance. This information is useful if the resonance frequency of a crystal is unknown. A brief frequency sweep is carried out until the phase comparator changes over and thus marks the resonance. For AT quartzes we know that the lowest usable frequency is the fundamental wave. The anharmonics are slightly above that. This information is not only important for the beginning, but also in the rare case that the instrument loses “track” of the fundamental wave. Once the frequency spectrum of the crystal is determined, the instrument must track the shift in resonance frequency, constantly carry out frequency measurements and then convert them into thickness.

Use of the “intelligent” measuring system has a number of obvious advantages over the earlier generation of active oscillators, primarily insensitivity

to mode hopping as well as speed and accuracy of measurement. This technique also enables the introduction of sophisticated properties which were not even conceivable with an active oscillator setup. The same device that permits the new technology to identify the fundamental wave with one sweep can also be used to identify other oscillation modes, such as the anharmonics or quasi-harmonics. The unit not only has a device for constantly tracking the fundamental wave, but can also be employed to jump back and forth between two or more modes. This query of different modes can take place for two modes with 10 Hz on the same crystal.

6.8 Auto Z match technique

The only catch in the use of equation 6.4 is that the acoustic impedance must be known. There are a number of cases where a compromise has to be made with accuracy due to incomplete or restricted knowledge of the material constants of the coating material:

1) The Z values of the solid material often deviate from those of a coating. Thin coatings are very sensitive to process parameters, especially in a sputter environment. As a result, the existing values for solid material are not adequate.

2) For many exotic substances, including alloys, the Z value is not known and not easy to determine.

3) It is repeatedly necessary to carry out a precise coating thickness measurement for multiple coating with the same crystal sensor. This applies in particular to optical multiple and semi-conductor coatings with a high temperature coefficient T_c. However, the effective Z value of the mixture of multiple coatings is unknown.

In such a case, therefore, the only effective method is to assume a Z value of 1, i.e. to ignore reality with respect to wave propagation in multi-substance systems. This incorrect assumption causes errors in the prediction of thickness and rate. The magnitude of the error depends on the coating thickness and the amount of deviation from the actual Z value.

In 1989 A. Wajid invented the **mode-lock** oscillator. He presumed that a connection existed between the fundamental wave and one of the anharmonics, similar to that ascertained by Benes between the fundamental oscillation and the third quasi-harmonic oscillation. The frequencies of the fundamental and the anharmonic oscillations are very similar and they solve the problem of the capacity of long cables. He found the necessary considerations for establishing this connection in works by Wilson (1954) as well as Tiersten and Smythe (1979).

The contour of the crystal, i.e. the spherical shape of one side, has the effect of separating the individual modes further from each other and preventing energy transfer from one mode to another. The usual method of identification is to designate the fundamental oscillation as (100), the lowest anharmonic frequency as (102) and the next higher anharmonic as (120). These three indices of the mode nomenclature are based on the number of phase reversals in the wave motion along the three crystal axes. The above mentioned works by Wilson, Tiersten and Smythe examine the properties of the modes by studying the influence of the radius of the cut on the position of the anharmonic in relation to the fundamental oscillation.

If one side of the quartz is coated with material, the spectrum of the resonances is shifted to lower frequencies. It has been observed that the three above mentioned modes have a somewhat differing mass sensitivity and thus experience somewhat different frequency shifts. This difference is utilized to determine the Z value of the material. By using the equations for the individual modes and observing the frequencies for the (100) and the (102) mode, one can calculate the ratio of the two elastic constants C_{60} and C_{55}. These two elastic constants are based on the shear motion. The key element in Wajid's theory is the following equation:

$$\frac{(C_{55}/C_{66})_{coated}}{(C_{55}/C_{66})_{uncoated}} \approx \frac{1}{(1+M\cdot Z)} \qquad (6.5)$$

with

M ... area mass/density ratio (ratio of coating mass to quartz mass per area unit)
Z ... Z value

It is a fortunate coincidence that the product M • Z also appears in the Lu-Lewis equation (equation 6.4). It can be used to assess the effective Z value from the following equations:

$$\tan\left(M\cdot Z\cdot\pi\cdot\frac{F_c}{F_q}\right)+Z\cdot\tan\left(\pi\cdot\frac{F_c}{F_q}\right)=0 \qquad (6.6)$$

or

$$Z=-\frac{\tan\left(M\cdot Z\cdot\pi\cdot\frac{F_c}{F_q}\right)}{\tan\left(\pi\cdot\frac{F_c}{F_q}\right)}$$

Here F_q and F_c are the frequencies of the non-coated or coated quartz in the (100) mode of the fundamental wave. Because of the ambiguity of the mathematical functions used, the Z value calculated in this way is not always a positively defined variable. This has no consequences of any significance because M is determined in another way by assessing Z and measuring the frequency shift. Therefore, the thickness and rate of the coating are calculated one after the other from the known M.

One must be aware of the limits of this technique. Since the assessment of Z depends on frequency shifts of two modes, any minimal shift leads to errors due to substantial mechanical or thermal stresses. It is not necessary to mention that under such circumstances the Z match technique, too, leads to similar errors. Nevertheless, the automatic Z value determination of the Z match technique is somewhat more reliable regarding occurrence of errors because the amplitude distribution of the (102) mode is asymmetric over the active crystal surface and that of the (100) mode is symmetric.

According to our experience, coating-related stresses have the most unfavorable effect on the crystal. This effect is particularly pronounced in the presence of gas, e.g. in sputter processes or reactive vacuum coating or sputter processes. If the Z value for solid material is known, it is better to use it than to carry out automatic determination of the "auto Z ratio". In cases of parallel coating and coating sequences, however, automatic Z determination is significantly better.

6.9 Coating thickness regulation

The last point to be treated here is the theory of the closed loop for coating thickness measuring units to effect coating growth at a controlled (constant) growth rate. The measuring advantages of the instruments, such as speed, precision and reliability, would not be completely exploited if this information were not inputted into an improved process monitoring system. For a coating process this means the coating rate should be kept as close and stable as possible to a setpoint. The purpose of the closed loop is to make use of the information flow of the measuring system in order to regulate the capacity for a special evaporation source in an appropriately adapted way. When the system functions correctly, the controller translates small deviations of the controlled parameter (the rate) from the setpoint into correction values of the re-adjusted evaporation capacity parameter. The ability of the controller to measure quickly and precisely keeps the process from deviating significantly from the setpoint.

The most widespread type of controller is the PID controller. Here **P** stands for proportional, **I** for integral and **D** for differential control function. In the following some of the properties of this controller are described in detail. Information on the system behavior is gained through a step response to a control fault in certain controller settings.

This response is recorded, and then improved control parameters for a new test are estimated. This procedure is continued until a satisfactory result is achieved. At the end the controller is optimized so that its parameters exactly match the characteristics of the evaporator source.

It is a long and frustrating process to adjust a controller to an evaporation source, requiring several minutes for stabilization and hours to obtain satisfactory results. Often the parameters selected for a certain rate are not suitable for an altered rate. Thus, a controller should ideally adjust itself, as the new controllers in INFICON coating measuring units do. At the beginning of installation and connection the user has the unit measure the characteristics of the evaporation source. Either a PID controller is used as the basis for slow sources or another type of controller for fast sources without significant dead time.

In relevant literature a distinction is made between three different ways of setting controllers. Depending on which data are used for the setting, a distinction is made between the closed loop, open loop and resonance response method.

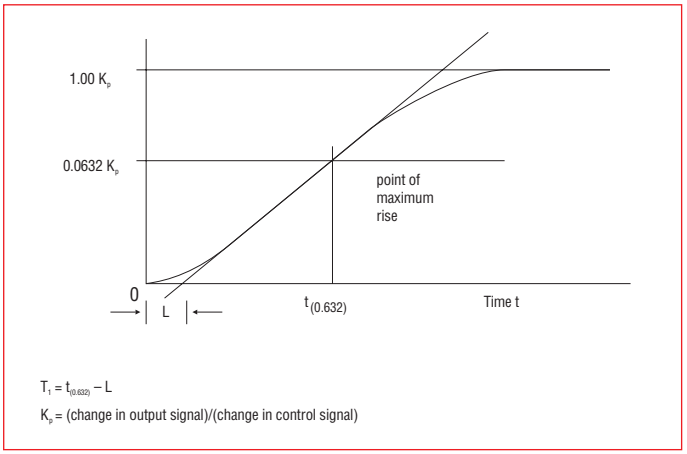


Fig. 6.8 Process response to a step change with t = 0 (open loop, control signal amplified)

Due to the simplicity with which the experimental data can be obtained, we preferred the open loop method. Moreover, application of this technique permits extensive elimination of the trial and error method.

The Auto Control Tune function developed by INFICON characterizes a process on the basis of its step responses. After a step-by-step change in the power the resulting changes in the rate as a function of time are smoothed and stored. The important step responses are determined, see Fig. 6.8.

In general, it is not possible to characterize all processes exactly, so several approximations have to be made. Normally one assumes that the dynamic characteristic can be reproduced by a process of the first order plus dead time. The Laplace transformation for this assumption (transfer to the s plane) is approximated:

$$\frac{\text{Output}}{\text{Input}} = \frac{K_p \cdot 10^{\frac{-L}{s}}}{\tau \cdot s + 1} \qquad \text{with(6.8)}$$

K_p = amplification in stationary state
L = dead time
τ = time constant

These three parameters are determined through the response curve of the process. An attempt has been made by means of several methods to calculate the required parameters of the system response from curves, as shown in Fig. 6.8. This results in a 1-point accordance at 63.2 % of the transition (a time constant), an exponential accordance at two points and an exponential accordance weighted according to the method of the smallest squares. A process is sufficiently characterized by this information so that the controller algorithm can be applied. Equation 6.9 shows the Laplace transformation for the very often used PID controller:

$$M(s) = K_c \cdot \left(1 + \frac{S}{T_i} + T_d \cdot S\right) \cdot E(s) \qquad (6.9)$$

with

M(s) = controlled variable or power
K_c = Control amplification (the proportional term)
T_i = integration time
T_d = differentiation time
E(s) = process deviation

Fig. 6.9 shows the control algorithm and a process with a phase shift of the first order and a dead time. The dynamics of the measuring device and the control elements (in our case the evaporator and the power supply) are

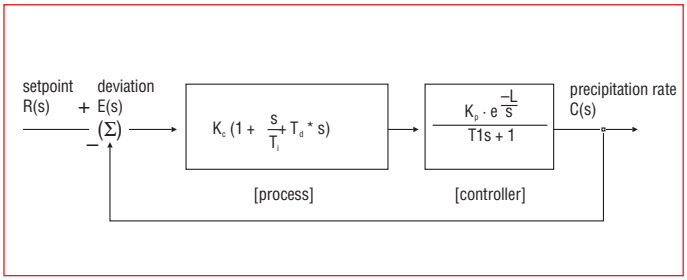


Fig. 6.9 Block diagram of the PID controller

implicitly contained in the process block. R(s) represents the rate setpoint. The return mechanism is the deviation created between the measured precipitation rate C(s) and the rate setpoint R(s).

The key to use of any control system is to select the correct values for K_c, T_d and T_i. The "optimum control" is a somewhat subjective term that is made clear by the presence of different mathematical definitions:

Usually the smallest square error ISE (Integral Square Error) is used as a measure of the quality of the control:

$$ISE = \int e^2(t) \cdot dt \qquad (6.10)$$

Here e is the error (the deviation): e = rate setpoint minus measured rate. ISE is relatively insensitive to small deviations, but large deviations contribute substantially to the value of the integral. The result is small "overshoots", but long ripple times because deviations occurring late contribute little to the integral.

The integral of the absolute value of the deviation IAE (Integral Absolute Error) was also proposed as a measure for control quality:

$$IAE = \int |e(t)| \cdot dt \qquad (6.11)$$

This is more sensitive for small deviations, but less sensitive for large deviations than ISE.

Graham and Lanthrop introduced the integral over time, multiplied by the absolute error ITAE (Integral Time Absolute Error), as a measure for control quality:

$$ITAE = \int t \cdot |e(t)| \cdot dt \qquad (6.12)$$

The ITAE is sensitive to initial and, to a certain extent, unavoidable deviations. Optimum control responses defined through ITAE consequently have short response times and larger "overshoots" than in the case of the other two criteria. However, ITAE has proven to be very useful for evaluating the regulation of coating processes.

INFICON's **Auto Control Tune** is based on measurements of the system response with an open loop. The characteristic of the system response is calculated on the basis of a step change in the control signal. It is determined experimentally through two kinds of curve accordance at two points. This can be done either quickly with a random rate or more precisely with a rate close to the desired setpoint. Since the process response depends on the position of the system (in our case the coating growth rate), it is best measured near the desired work point. The process information measured in this way (process amplification K_p, time constant T_1 and dead time L) are used to generate the most appropriate PID control parameters.

The best results in evaluating coating control units are achieved with ITAE. There are overshoots, but the reaction is fast and the ripple time short. Controller setting conditions have been worked up for all integral evaluation criteria just mentioned so as to minimize the related deviations. With a manual input as well as with experimental determination of the process response coefficients, the ideal PID coefficients for the ITAE evaluation can easily be calculated from equations 6.13, 6.14 and 6.15:

$$K_c = \left(\frac{1.36}{K_p}\right) \cdot \left(\frac{L}{T_1}\right)^{-0.947} \qquad (6.13)$$

$$T_i = \left(\frac{1.19}{T_1}\right) \cdot \left(\frac{L}{T_1}\right)^{0.738} \qquad (6.14)$$

$$T_d = (0.381 \cdot T_1) \cdot \left(\frac{L}{T_1}\right)^{0.995} \qquad (6.15)$$

For slow systems the time interval between the forced changes in control voltage is extended to avoid "hanging" the controller (hanging = rapid growth of the control signal without the system being able to respond to the altered signal). This makes a response to the previous change in the controller setting and "powerful" controller settings possible. Another advantage is the greater insensitivity to process noise because the data used for control do not come from merely one measurement, but from several, so that the mass-integrating nature of the quartz crystal is utilized.

In processes with short response times (short time constants) and small to unmeasurable dead times, the PID controller often has difficulties with the noise of the coating process (beam deflection, rapid thermal short-circuits between melt and evaporator, etc.). In these cases a control algorithm of the integral reset type is used with success. This controller always integrates the deviation and presses the system towards zero deviation. This technique works well with small or completely imperceptible dead times. However, if it is used with a noticeable phase shift or dead time, the controller tends to generate oscillations because it overcompensates the controller signal before the system has a chance to respond. **Auto Control Tune** recognizes the properties of these fast systems during the measurement of a step response and utilizes the information to calculate the control amplification for a non-PID control algorithm.

6.10 INFICON instrument variants

The instrument models available differ both in hardware and software equipment: the simplest unit, the XTM/2, is purely a measuring or display device that cannot control vacuum coating.

The XTC/2 and XTC/C group can control vacuum coating sources and up to three different coatings of a process (not to be confused with nine different coating programs). In the case of XTM/2, XTC/2 and XTC/C units, the AutoZero and AutoTune functions are not available, and measurement with several sensors simultaneously as well as simultaneous control of two vacuum coating sources are not possible.

However, the IC/5 offers all comfort functions available today: measurement with up to eight sensors with AutoZero and AutoTune as well as capability of simultaneous control of two evaporator sources. Moreover, it offers 24 material programs, with which 250 coatings in 50 processes can be programmed. To simplify operation and avoid errors, the unit also has a diskette drive. All types of crystal holders can be connected here. The thickness resolution is around 1 Å, the rate resolution for rates between 0 and 99.9 Å/s around 0.1 Å/s and for rates between 100 and 999 Å/s around 1 Å/s. A particularly attractive option offered by the IC/5 is a microbalance

Thin film controllers/control units

board with a highly stable reference quartz. This oscillator is 50 times more stable than the standard oscillator; long-term stability and accuracy are then 2 ppm over the entire temperature range. This option is specially designed for coatings of material with low density and at low coating rates. This is important for space contamination and sorption studies, for example.

7. Application of vacuum technology for coating techniques

7.1 Vacuum coating technique

Vacuum technology has been increasingly used in industrial production processes during the last two decades. Some of these processes and their typical working pressure ranges are shown in Fig. 7.1.

Since a discussion of all processes is beyond the scope of this brochure, this section will be restricted to a discussion of several examples of applications in the important field of coating technology.

Deposition of thin films is used to change the surface properties of the base material, the substrate. For example, optical properties such as transmission or reflection of lenses and other glass products, can be adjusted by applying suitable coating layer systems. Metal coatings on plastic web produce conductive coatings for film capacitors. Polymer layers on metals enhance the corrosion resistance of the substrate.

Through the use of vacuum it is possible to create coatings with a high degree of uniform thickness ranging from several nanometers to more than 100 mm while still achieving very good reproducibility of the coating properties. Flat substrates, web and strip, as well as complex molded-plastic parts can be coated with virtually no restrictions as to the substrate material. For example, metals, alloys, glass, ceramics, plastics and paper can be coated. The variety of coating materials is also very large. In addition to metal and alloy coatings, layers may be produced from various chemical compounds or layers of different materials applied in sandwich form. A significant advantage of vacuum coating over other methods is that many special coating properties desired, such as structure, hardness, electrical conductivity or refractive index, are obtained merely by selecting a specific coating method and the process paramaters for a certain coating material.

7.2 Coating sources

In all vacuum coating methods layers are formed by deposition of material from the gas phase. The coating material may be formed by physical processes such as evaporation and sputtering, or by chemical reaction. Therefore, a distinction is made between physical and chemical vapor deposition:

- physical vapor deposition = PVD
- chemical vapor deposition = CVD.

7.2.1 Thermal evaporators (boats, wires etc.)

In the evaporation process the material to be deposited is heated to a temperature high enough to reach a sufficiently high vapor pressure and the desired evaporation or condensation rate is set. The simplest sources used in evaporation consist of wire filaments, boats of sheet metal or electrically conductive ceramics that are heated by passing an electrical current through them (Fig. 7.2). However, there are restrictions regarding the type of material to be heated. In some cases it is not possible to achieve the

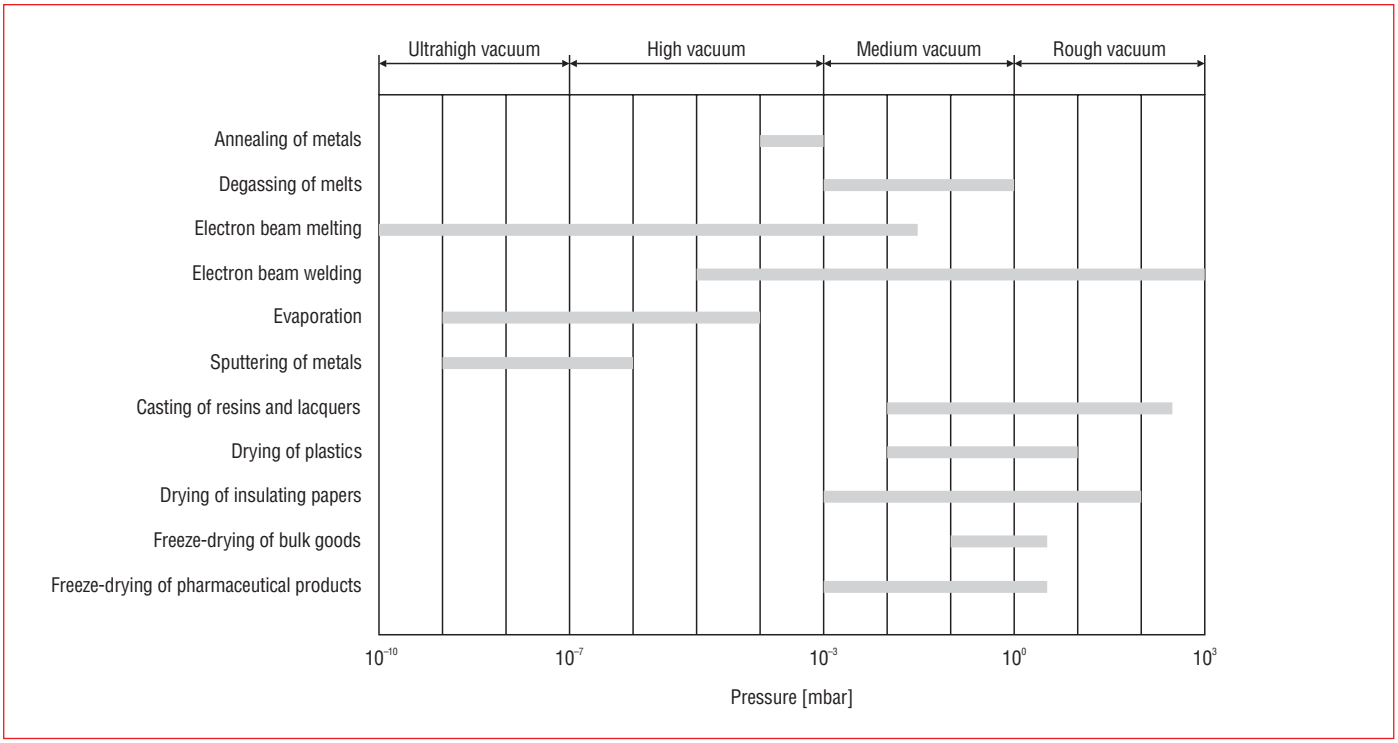


Fig. 7.1 Pressure ranges for various industrial processes

Applications of vacuum technology

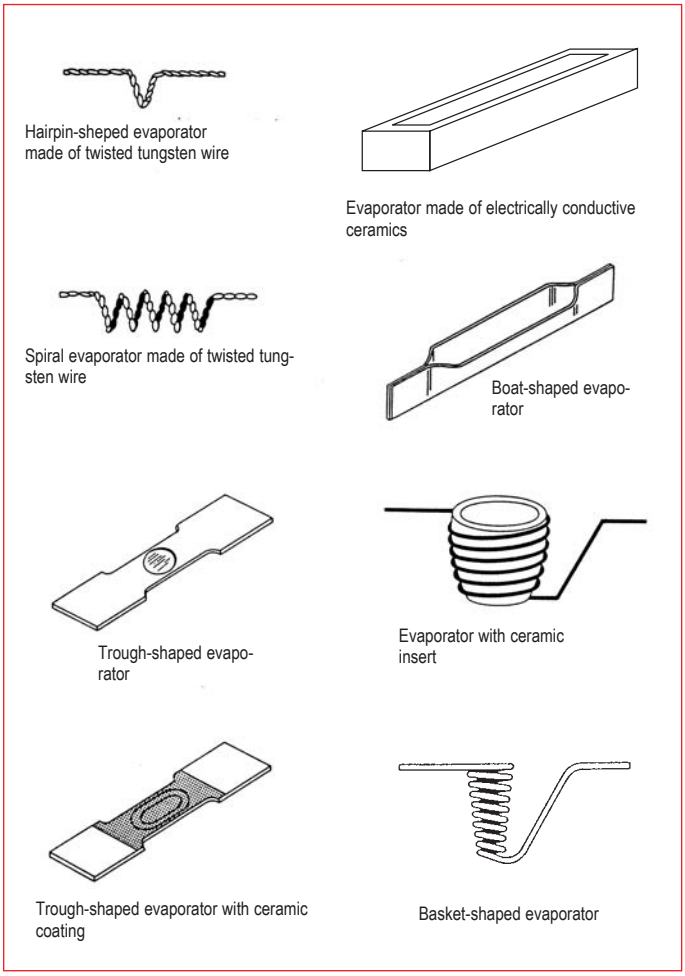
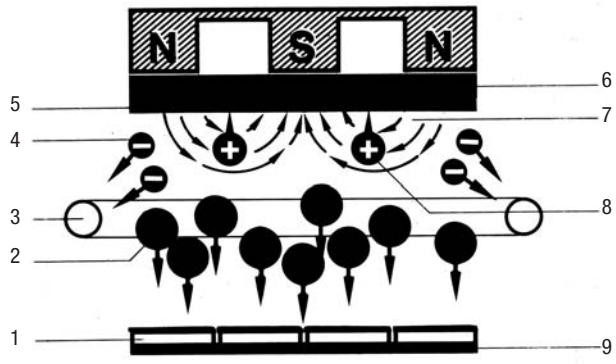


Fig. 7.2 Various thermal evaporators

necessary evaporator temperatures without significantly evaporating the source holder and thus contaminating the coating. Furthermore chemical reactions between the holder and the material to be evaporated can occur resulting in either a reduction of the lifetime of the evaporator or contamination of the coating.

7.2.2 Electron beam evaporators (electron guns)

To evaporate coating material using an electron beam gun, the material, which is kept in a water-cooled crucible, is bombarded by a focused electron beam and thereby heated. Since the crucible remains cold, in principle, contamination of the coating by crucible material is avoided and a high degree of coating purity is achieved. With the focused electron beam, very high temperatures of the material to be evaporated can be obtained and thus very high evaporation rates. Consequently, high-melting point compounds such as oxides can be evaporated in addition to metals and alloys. By changing the power of the electron beam the evaporation rate is easily and rapidly controlled.



1 Substrates
2 Sputtered atoms
3 Anode
4 Electrons
5 Target
6 Cathode
7 Magnetic field lines
8 Argon ions
9 Substrate

Fig. 7.3 Schematic diagram of a high-performance cathode sputter arrangement

7.2.3 Cathode sputtering

In the cathode sputtering process, the target, a solid, is bombarded with high energy ions in a gas discharge (Fig. 7.3). The impinging ions transfer their momentum to the atoms in the target material, knocking them off. These displaced atoms – the sputtered particles – condense on the substrate facing the target. Compared to evaporated particles, sputtered particles have considerably higher kinetic energy. Therefore, the conditions for condensation and layer growth are very different in the two processes. Sputtered layers usually have higher adhesive strength and a denser coating structure than evaporated ones. Sputter cathodes are available in many different geometric shapes and sizes as well as electrical circuits configurations. What all sputter cathodes have in common is a large particle source area compared to evaporators and the capability to coat large substrates with a high degree of uniformity. In this type of process metals, alloys of any composition as well as oxides can be used as coating materials.

7.2.4 Chemical vapor deposition

In contrast to PVD methods, where the substance to be deposited is either solid or liquid, in chemical vapor deposition the substance is already in the vapor phase when admitted to the vacuum system. To deposit it, the substance must be thermally excited, i.e. by means of appropriate high temperatures or with a plasma. Generally, in this type of process, a large number of chemical reactions take place, some of which are taken advantage of to control the desired composition and properties of the coating. For example, using silicon-hydrogen monomers, soft Si-H polymer coatings, hard silicon coatings or – by addition of oxygen – quartz coatings can be created by controlling process parameters.

7.3 Vacuum coating technology/ coating systems

7.3.1 Coating of parts

For molded-plastic parts, vacuum coating techniques are increasingly replacing conventional coating methods, such as electroplating. For example, using vacuum coating methods, automobile reflectors obtain a mirror-like surface, plastic articles in the furniture, decoration, clock and watch as well as electronics industry are metal-coated and optical effects are created on articles in the decoration industry.

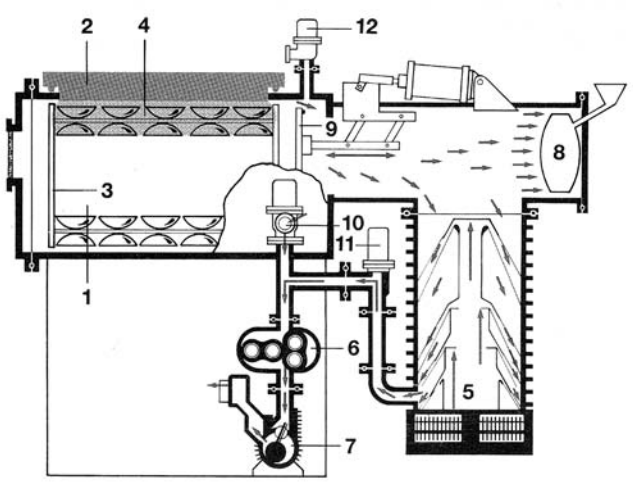
Fig. 7.4 shows a type of vacuum system in which large batches of molded-plastic parts can be coated simultaneously. The substrates are placed on a cage that rotates past the coating source, a sputter cathode in this example. In some applications, by using a glow discharge treatment, the substrates are cleaned and the surface is activated prior to the coating process. This enhances the adhesive strength and reproducibility of the coating properties. A corrosion protection coating can be applied after sputtering. In this case, a monomer vapor is admitted into the system and a high-frequency plasma discharge ignited. The monomer is actived in the plasma and deposits on the substrates as a polymer coating. In this type of system there may be plastic substrates with a surface area of several 10 m^2 on the cage, causing a correspondingly high desorption gas flow. The vacuum system must be able to attain the required pressures reliably despite these high gas loads. In the example shown, the system is evacuated with a combination of a backing and Roots pump. A diffusion pump along with a cold surface forms the high vacuum pump system. The cold surfaces pump a large portion of the vapor and volatile substances emitted by the plastic parts while the diffusion pump basically removes the non-condensable gases as well as the noble gas required for the sputter process.

A completely different concept for the same process steps is shown in Fig. 7.5. The system consists of four separate stations made up of a drum rotating around the vertical axis with four substrate chambers and process stations mounted in the vacuum chamber. During rotation, a substrate chamber moves from the loading and unloading station to the pretreatment station, to the metallization station, to the protective coating station and then back to the initial position. Since each station has its own pumping system, all four processes can run simultaneously with entirely independent adjustable process parameters. The vacuum system comprises of turbomolecular pumps and backing pump sets consisting of Roots and rotary vane pumps.

7.3.2 Web coating

Metal-coated plastic webs and papers play an important role in food packaging. They preserve food longer according to storage and transport logistics requirements and give packaging an attractive appearence. Another important area of application of metal-coated web is the production of film capacitors for electrical and electronics applications. Metal-coating is carried out in vacuum web coating systems. Fig. 7.6 shows a typical scheme. The unit consists of two chambers, the winding chamber with the roll of web to be coated and the winding system, as well as the coating chamber, where the evaporators are located. The two chambers are sealed from each other, except for two slits through which the web runs. This makes it possible to pump high gas loads from the web roll using a relatively small pumping set. The pressure in the winding chamber may be more than a factor of 100 higher than the pressure simultaneously established in the coating chamber. The pump set for the winding chamber usually consists of a combination of Roots and rotary vane pumps.

With strongly degassing rolls of paper, it may be necessary to install a cold surface in the winding chamber to act as a water vapor pump. The rolls of the plastic web or paper typically have diameters between 400 and 1000 mm and a width of 400 to 3000 mm. A precise, electronically controlled winding system is required for winding and unwinding as well as web guidance.



1 Vacuum chamber
2 High-performance cathode
3 Substrate holder
4 Substrates
5 Diffusion pump
6 Roots pump
7 Rotary piston pump
8 Cold trap
9 High vacuum valve
10 Valve for bypass line
11 Foreline valve
12 Venting valve

Fig. 7.4 Diagramm of a batch system for coating parts

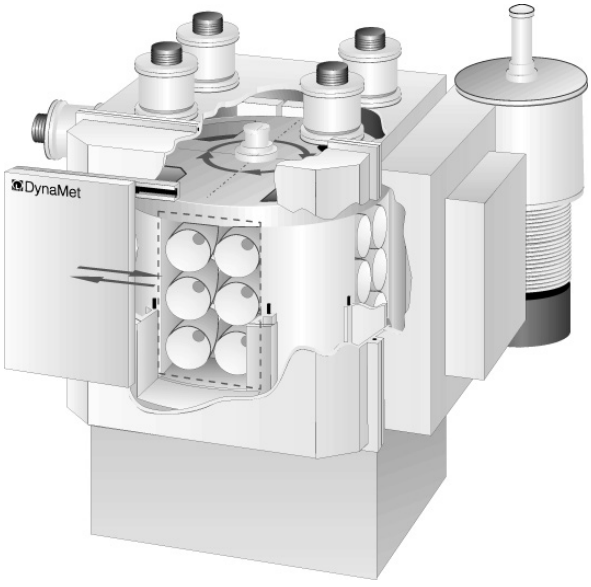
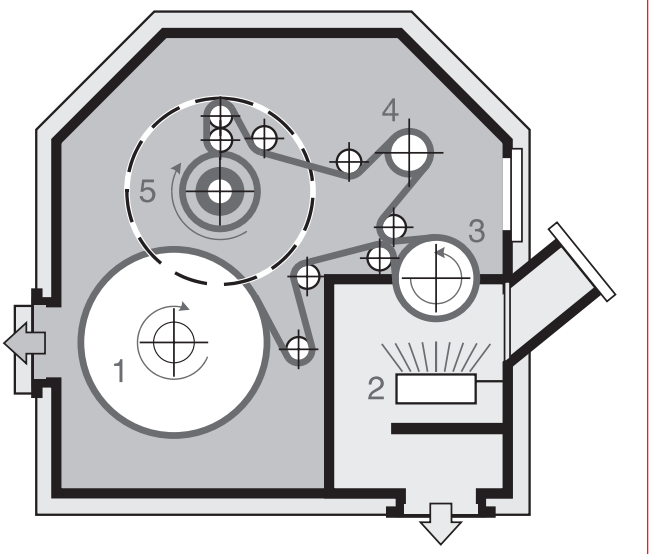


Fig. 7.5 Multi-chamber parts-coating unit (rotationally symmetric in-line system DynaMet 4V)

Applications of vacuum technology



1 Unwinder
2 Coating source
3 Coating roller
4 Drawing roller
5 Take-up reel

Fig. 7.6 Schematic diagram of a vacuum web coating system

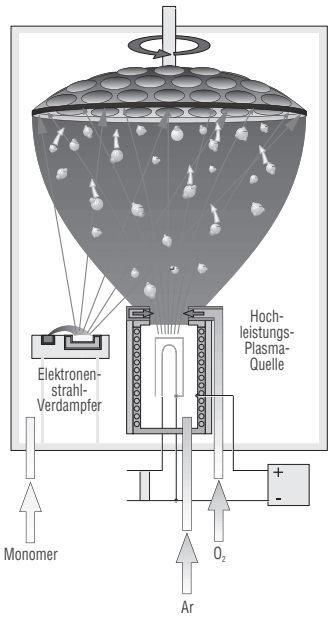


Fig. 7.7 Coating unit for optical coating systems

During the coating process the web, at a speed of more than 10 m/s, passes a group of evaporators consisting of ceramic boats, from which aluminium is evaporated. To achieve the necessary Al-coating thickness at these high web speeds, very high evaporation rates are required. The evaporators must be run at temperatures in excess of 1400 °C. The thermal radiation of the evaporators, together with the heat of condensation of the growing layer, yields a considerable thermal load for the web. With the help of cooled rollers, the foil is cooled during and after coating so that it is not damaged during coating and has cooled down sufficiently prior to winding.

During the entire coating process the coating thickness is continuously monitored with an optical measuring system or by means of electrical resistance measurement devices. The measured values are compared with the coating thickness setpoints in the system and the evaporator power is thus automatically controlled.

7.3.3 Optical coatings

Vacuum coatings have a broad range of applications in production of ophthalmic optics, lenses for cameras and other optical instruments as well as a wide variety of optical filters and special mirrors. To obtain the desired transmission or reflection properties, at least three, but sometimes up to 50 coatings are applied to the glass or plastic substrates. The coating properties, such as thickness and refractive index of the individual coatings, must be controlled very precisely and matched to each other. Most of these coatings are produced using electron beam evaporators in single-chamber units (Fig. 7.7). The evaporators are installed at the bottom of the chamber, usually with automatically operated crucibles, in which there are several different materials. The substrates are mounted on a rotating calotte above the evaporators. Application of suitable shieldings combined with relative movement between evaporators and substrates, results in a very high degree of coating uniformity. With the help of quartz coating thickness monitors (see Section 6) and direct measurement of the attained optical properties of the coating system during coating, the coating process is fully controlled automatically.

One of the key requirements of coatings is that they retain their properties under usual ambient conditions over long periods of time. This requires to produce the densest coatings possible, into which neither oxygen nor water can penetrate. Using glass lenses, this is achieved by keeping the substrates at temperatures up to 300 °C during coating by means of radiation heaters. However, plastic lenses, as those used in eyeglass optics, are not allowed to be heated above 80 °C. To obtain dense, stable coatings these substrates are bombarded with Ar ions from an ion source during coating. Through the ion bombardement the right amount of energy is applied to the growing layer so that the coated particles are arranged on the energetically most favorable lattice sites, without the substrate temperature reaching unacceptably high values. At the same time oxygen can be added to the argon. The resulting oxygen ions are very reactive and ensure that the oxygen is included in the growing layer as desired.

The vacuum system of such a coating unit usually consists of a backing pump set comprising a rotary vane pump and Roots pump as well as a high vacuum pumping system. Depending on the requirements, diffusion pumps, cryo pumps or turbomolecular pumps are used here, in most cases in connection with large refrigerator-cooled cold surfaces. The pumps must be installed and protected by shieldings in a way that no coating material can enter the pumps and the heaters in the system do not thermally overload them. Since shielding always reduces the effective pumping speed, the system manufacturer must find a suitable compromise between shielding effect and reduction of pumping speed.

7.3.4 Glass coating

Coated glass plays a major role in a number of applications: window panes in moderate and cold climate zones are provided with heat-reflecting coating systems to lower heating costs; in countries with high intensity solar radiation, solar protection coatings are used that reduce air conditioning costs; coated car windows reduce the heating-up of the interior and mirrors are used both in the furniture and the automobile industry. Most of these coatings are produced in large in-line vacuum systems. Fig. 7.8 shows a typical system. The individual glass panes are transported into a entrance chamber at atmospheric pressure. After the entrance valve is closed, the chamber is evacuated with a forepump set. As soon as the pressure is low enough, the valve to the evacuated transfer chamber can be opened. The glass pane is moved into the transfer chamber and from there at constant speed to the process chambers, where coating is carried out by means of sputter cathodes. On the exit side there is, in analogy to the entrance side, a transfer chamber in which the pane is parked until it can be transferred out through the exit chamber.

Most of the coatings consist of a stack of alternative layers of metal and oxide. Since the metal layers may not be contaminated with oxygen, the individual process stations have to be vacuum-isolated from each other and from the transfer stations. Utilization of valves for separating process chambers is unsatisfactory because it increases plant dimensions. To avoid frequent and undesirable starting and stopping of the glass panes, the process chambers are vacuum-separated through so-called “slit locks”, i.e. constantly open slits combined with an intermediate chamber with its own vacuum pump (Fig. 7.9). The gaps in the slits are kept as small as technically possible to minimize clearance and therefore conductance as the glass panes are transported through them. The pumping speed at the intermediate chamber is kept as high as possible in order to achieve a considerably lower pressure in the intermediate chamber than in the process chambers. This lower pressure greatly reduces the gas flow from a process chamber via the intermediate chamber to the adjacent process chamber. For very stringent separation requirements it may be necessary to place several intermediate chambers between two process chambers.

The glass coating process requires high gas flows for the sputter processes as well as low hydrocarbon concentration. The only vacuum pump which satisfies these requirements as well as high pumping speed stability over time are turbo-molecular pumps which are used almost exclusively.

While the transfer and process chambers are constantly evacuated, the entrance and exit chambers must be periodically vented and then evacuated again. Due to the large volumes of these chambers and the short cycle times, a high pumping speed is required. It is provided by combinations of rotary vane pumps and Roots pumps. For particularly short cycle times gas cooled Roots pumps are also used.

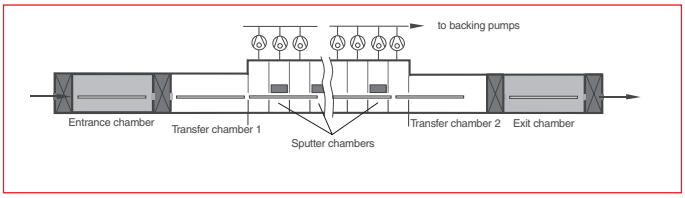


Fig. 7.8 Plant for coating glass panes – 3-chamber in-line system, throughput up to 3,600,000 m^2 / year

All major functions of a plant, such as glass transport, control of the sputter processes and pump control, are carried out fully automatically. This is the only way to ensure high productivity along with high product quality.

7.3.5 Systems for producing data storage disks

Coatings for magnetic- or magneto-optic data storage media usually consist of several functional coatings that are applied to mechanically finished disks. If several plates are placed on one common carrier, the coating processes can be carried out in a system using a similar principle to that used for glass coating. However, most disks must be coated on both sides and there are substantially greater low particle contamination requirements as compared to glass coating. Therefore, in-line systems for data memories use a vertical carrier that runs through the system (Fig. 7.10). The sputter cathodes in the process stations are mounted on both sides of the carrier so that the front and back side of the disk can be coated simultaneously.

An entirely different concept is applied for coating of single disks. In this case the different process stations are arranged in a circle in a vacuum chamber (Fig. 7.11). The disks are transferred individually from a magazine to a star-shaped transport arm. The transport arm cycles one station further after each process step and in this way transports the substrates from one process station to the next. During cycling all processes are switched off and the stations are vacuum-linked to each other. As soon as the arm has reached the process position, the individual stations are seperated from each other by closing seals. Each station is pumped by means of its own turbomolecular pump and the individual processes are started. As many process stations as there are in the system as many processes can be performed in parallel. By sealing off the process stations, excellent vacuum separation of the individual processes can be achieved. However, since the slowest process step determines the cycle interval, two process stations may have to be dedicated for particularly timeconsuming processes.

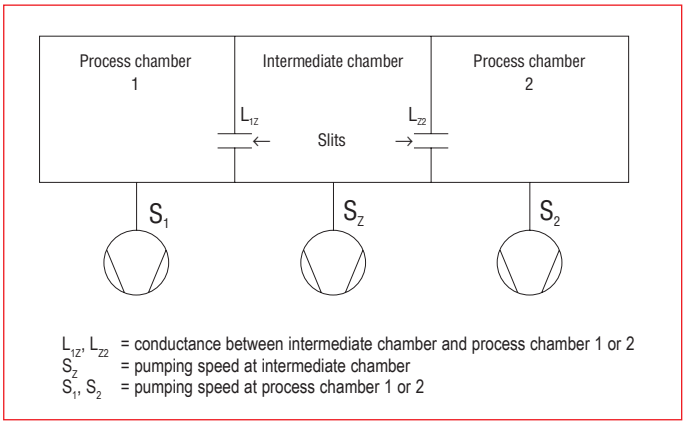


Fig. 7.9 Principle of chamber separation through pressure stages

Applications of vacuum technology

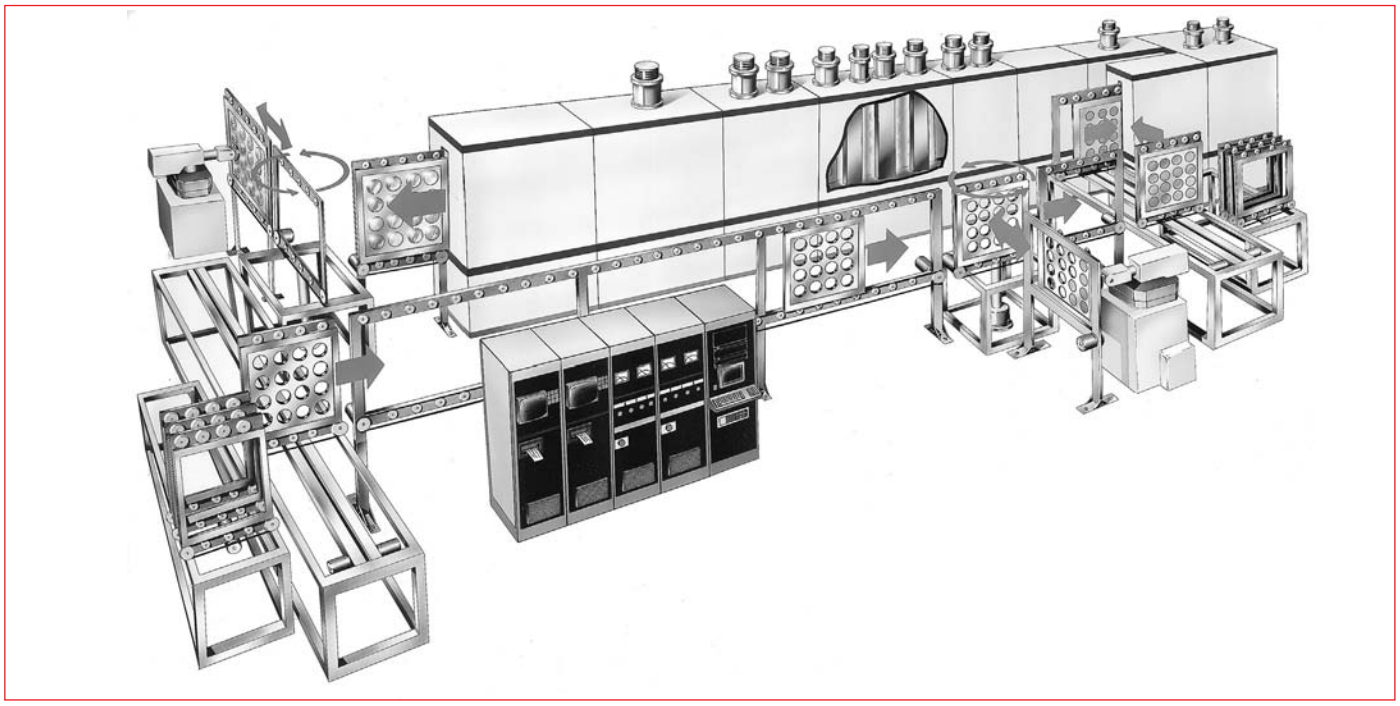


Fig. 7.10 Plant for coating data storage disks with carrier transport system

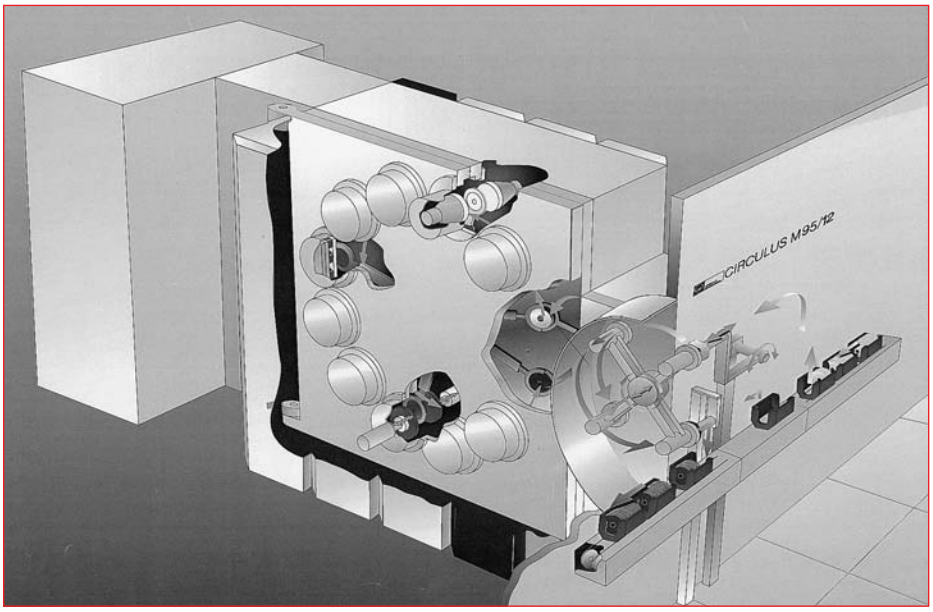


Fig. 7.11 Plant for individual coating of data storage disks

8. Instructions for vacuum equipment operation

8.1 Causes of faults where the desired ultimate pressure is not achieved or is achieved too slowly

If the desired ultimate pressure is not reached at all in vacuum equipment or if it is attained only after an excessively long pumping period, then the following problems may be the reason:

If the desired ultimate pressure is not reached, then

- the apparatus may be leaky or dirty,
- the pump may dirty or damaged,
- the vacuum gauge may be defective.

If the desired ultimate pressure is reached only after a very long running time, then

- the apparatus may be dirty,
- the pumping line may be restricted,
- the pump may be dirty or of too small a capacity,
- the pumping speed may be restricted due to other causes.

In order to locate the fault, one normally proceeds by separating the evacuated vessel from the pump system (where this is possible) and checking the vessel alone for leaks and contamination using the pressure rise method, for example. If it has been found that the vessel is free of defects in this regard, then the measurement system will be checked for cleanliness (see Section 8.38) and ultimately – if required – the pump or the pumping system itself will be examined.

8.2 Contamination of vacuum vessels and eliminating contamination

In addition to the pressure rise method (Section 6.1) there is a further method for detecting contamination, based on the fact that condensable vapors will generally account for the major share of the gas mix in dirty vessels: here the pressure reading at a compression vacuum gauge (partial pressure for the non-condensable gases) is compared with that at an electrical vacuum gauge, e.g. a thermal conductivity or ionization vacuum gauge (measuring total pressure). These two vacuum gauges must, however, be clean themselves. Where vapors are present the compression vacuum gauge will indicate much better pressure than the electrical vacuum gauge. This is a sure sign that the vessel walls are contaminated, usually with oil. Another commonly used procedure is to compare the pressure indication of one and the same vacuum gauge (not a compression vacuum gauge) with and without a cold trap inserted in the line: Filling the cold trap with liquid nitrogen will cause the pressure to drop abruptly, by one power of ten or more, if the container is contaminated since the vapors will freeze out in the trap.

Eliminating contamination for glass equipment
If the contaminants exhibit a high vapor pressure, then they can be pumped out relatively quickly. If this is not successful, then the apparatus will have to be cleaned. Contaminated glass components will first be cleaned with chromic-sulfuric acid mixture or – if this is necessary – with dilute hydrofluoric acid (1:30). They are then rinsed with distilled water. If this does not bring about the desired results, then an organic solvent can be tried. Then the glass components will again have to be rinsed with methanol and distilled water. (Do not use denatured alcohol!)

Eliminating contamination at metallic equipment
Metal components will usually exhibit traces of contamination by oil and greases. If these cannot be readily removed by pumping down the vessel, then an appropriate organic solvent (denatured alcohol is unsuitable in all cases) will have to be used for cleaning. Maximum cleanliness can be achieved with vapor baths such as those commonly found in industry. If one desires to get down to extremely low pressure ranges ($< 10^{-7}$ mbar), then – after cleaning – the metal components will have to be baked out at temperatures of up to 200 °C. Seriously contaminated metal components will first have to be cleaned by cutting away or sandblasting the top surface. These methods suffer the disadvantage that the surface area for the surface thus treated will be increased through roughening and active centers may potentially be formed which would readily adsorb vapor molecules. Supplementary cleaning in the vapor bath (see above) is advisable. In some cases electrolytic pickling of the surface may be beneficial. In the case of high vacuum components it is necessary to pay attention to ensuring that the pickling does not turn into etching, which would seriously increase the surface area. Polishing surfaces which have been sandblasted is not necessary when working in the rough and medium vacuum ranges since the surface plays only a subordinate role in these pressure regimes.

8.3 General operating information for vacuum pumps

If no defects are found in the vacuum vessels and at the measurement tubes or if the apparatus still does not operate satisfactorily after the faults have been rectified, then one should first check the flange seals at the pump end of the system and possibly the shut-off valve. Flange seals are known to be places at which leaks can appear the most easily, resulting from slight scratches and mechanical damage which initially appears insignificant. If no defect can be discerned here, either, then it is advisable to check to see whether the pumps have been maintained in accordance with the operating instructions.

Given initially in this section are general instructions on pump maintenance, to be followed in order to avoid such defects from the very outset. In addition, potential errors and their causes are discussed.

Instructions for equipment operation

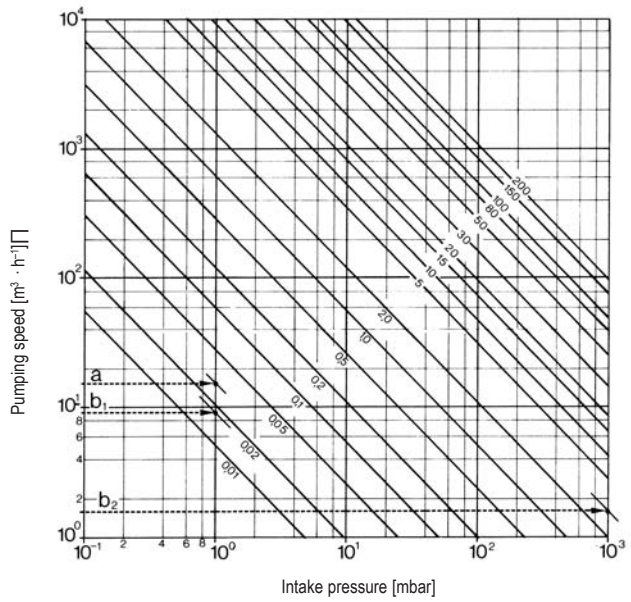
8.3.1 Oil-sealed rotary vacuum pumps (Rotary vane pumps and rotary piston pumps)

8.3.1.1 Oil consumption, oil contamination, oil change

The oil serves to:

- lubricate moving parts,
- seal moving parts against atmospheric pressure,
- seal the valve,
- fill the dead space below the valve,
- to seal the various operational spaces one from another.

In all pumps it is possible to check the oil charge during operation using the built-in oil level sight glass. During continuous operation in particular it is necessary to ensure that the oil charge never falls below the minimum level. During a pumping process oil-sealed rotary pumps will emit oil vapors from the discharge port, this being due to the high operating temperature. This leads to oil loss to an extent which will depend on the quantity of gas or vapor which is drawn into the pump. Larger oil droplets can be retained by installing a coarse separator in the discharge line. This will reduce oil loss considerably. The fine oil mist filter installed in some pumps will also retain the finest oil droplets (fine separation) so that no oil at all will leave the outlet of the pump and oil loss is reduced practically to zero since – as in coarse separation – the oil which is separated out is returned to the pump. With pumps without an integral fine separator this device is offered as an optional extra.



Example: Oil losses for a TRIVAC S 16 A at pressure of 1 mbar:
a) Without gas ballast: in accordance with the pumping speed curve S = 15 m³ / h; according to diagram: oil loss = 0.03 cm³ / h (line a)
b) With gas ballast: S = 9 m3/h at 1 mbar; oil loss = 0.018 cm³ / h (line b_1), plus additional loss due to gas ballast quantity (0.1 times the 1.6 m³ / h rated pumping speed); that is: Chart on the horizontal line b_2 up to atmospheric pressure: additional loss: 3 cm³ / h (diagonal line). Overall loss during gas ballast operation 0.018 + 3 = 3.018 cm³ / h.

Fig. 8.1 Oil loss for oil-sealed pumps (referenced to an approximate maximum value of 2 cm³ oil loss per cubic meter of air drawn in [STP])

If an oil-filled rotary pump is operated without an oil separation and return device, then it will be necessary to expect a certain amount of oil consumption, the extent of which will depend on the size of the pump and the nature of the operations. In the worst case this can amount to about 2 cm3 for every cubic meter of air pumped (at STP and including the gas ballast also drawn in). Figure 8.1 makes it possible to predict the amount of oil loss to be expected in practical situations. The example demonstrates that greater oil losses must be expected when operating the pump with gas ballast. This situation, which is generally valid, is always to be taken into account in practice.

If the pump oil has become unusabledue to exposure to the vapors or contaminants which are encountered in the process, then the oil will have to be replaced. It is impossible to formulate any hard-and-fast rules as to when an oil change will be required since the nature of the operations will determine how long the oil will remain good. Under clean conditions (e.g. backing pumps for diffusion pumps in electro-nuclear accelerators) rotary vacuum pumps can run for years without an oil change. Under extremely “dirty” conditions (e.g. during impregnation) it may be necessary to change the oil daily. The oil will have to be replaced when its original light brown color, has turned dark brown or black due to ageing or has become cloudy because liquid (such as water) has entered the pump. An oil change is also necessary when flakes form in corrosion protection oil, indicating that the corrosion protection agent is exhausted.

Changing the oil

The oil change should always be carried out with the pump switched off but at operating temperature. The oil drain (or fill) opening provided for each pump is to be used for this purpose. Where the pump is more seriously contaminated, then it should be cleaned. The applicable operating instructions are to be observed in this case.

8.3.1.2 Selection of the pump oil when handling aggressive vapors

If corrosive vapors (e.g. the vapors formed by acids) are to be pumped, then a PROTELEN® corrosion protection oil should be used in place of the normal pump oil (N 62). These types of vapors will then react with the basic (alkaline) corrosion protection agent in the oil. The continuous neutralization reactions will exhaust the corrosion protection agent at a rate depending on the quantity and acidity of the vapors. The oil will have to be changed more frequently, in accordance with these factors. Corrosion protection oils are either very hygroscopic or they easily form emulsions with water. Consequently a pump which is filled with corrosion protection oil will absorb moisture from the air if it is out of service for an extended period of time. In no case should one ever use a pump filled with corrosion protection oil in order to pump water vapor since the lubricating and corrosion inhibition properties of the oil would be adversely affected. Once the oil has absorbed water it will no longer be possible for such pumps to achieve the ultimate pressures which would be the case with fresh corrosion protection oil or standard pump oil (N 62). Oil-filled pumps should, under normal operating con-ditions, not be filled with corrosion protection oil. N 62 oil is preferred when pumping air, water vapor and non-corrosive organic vapors in so far as there is positive protection against the vapors condensing inside the pump.

8.3.1.3 Measures when pumping various chemical substances

This discussion cannot provide exhaustive coverage of the many and varied application fields for oil-filled vacuum pumps in the chemicals industry. Our many years of experience with the most difficult of chemicals applications can be used to solve your particular problems. Three aspects should, however, be mentioned briefly: pumping explosive gas mixes, condensable vapors, and corrosive vapors and gases.

Explosion protection
Applicable safety and environmental protection regulations shall be observed when planning and engineering vacuum systems. The operator must be familiar with the substances which the system will be pumping and take into account not only normal operating conditions but also abnormal situations, operating outside normal parameters. The most important aids to avoiding explosive mixtures are – in addition to inertization by adding protective gases – maintaining the explosion limit values with the aid of condensers, adsorption traps and gas scrubbers.

Protection against condensation
LEYBOLD pumps offer three options for keeping vapors from condensing in the pumps:

- The gas ballast principle (See Fig. 2.14). This increases considerably the amount of vapor which the pump can tolerate.
- Increased pump temperature. The rugged design of our pumps makes it possible to run them at temperatures of up to 120 °C. Thus the tolerance for pure water vapor, for example, will rise by a factor of five when compared with normal gas ballast operation.
- Using vacuum condensers (see Section 2.15). These act as selective pumps and should be sized so that the downstream gas ballast pump will not receive more vapor than the amount corresponding to the appropriate vapor tolerance.

Corrosion protection
Oil-sealed pumps are already quite satisfactorily protected against corrosion due to the oil film which will be present on all the component surfaces. Corrosion is defined here as the electrochemical dissolution of metals, i.e. the release of electrons by the metal atom and their acceptance by the oxidation agent (corrosive gas). A metal atom which is susceptible to corrosion must therefore be exposed to an active atom of the oxidation agent.

This makes clear how the oil-sealed pump is protected against corrosion; the concentration of the oxidation agent in the oil is negligible and thus the opportunity for the metal to release electrons is equally small. This also makes it clear that the use of so-called “non-rusting” or “stainless” steels does not make sense since oxidation is necessary for the passivation of these steels, in order to reach the so-called passive region for these steel compounds. The critical passivation current density will normally not appear in oil-sealed pumps.

a) **Acids**
Our pumps are fundamentally suited to pumping acids. In special situations problems with the oil and with auxiliary equipment attached at the intake and/or discharge end may occur. Our engineers in Cologne are available to assist in solving such problems.

b) **Anhydrides**
CO (carbon monoxide) is a strong reducing agent. When CO is being pumped it is therefore important that air not be used as the gas ballast but rather that inert gases be used at the very outside (e.g. Ar or N_2). Inert gas ballast should also be used when pumping SO_2, SO_3, and H_2S. A corrosion inhibiting oil is also to be used when handling these three anhydrides. Carbon dioxide (CO_2) can be pumped without making any special arrangements.

c) **Alkaline solutions**
Normal N 62 pump oil is to be used to pump basic (alkaline) solutions. Sodium hydroxide and caustic potash solutions should not be pumped in their concentrated form. Ammonia is highly amenable to pumping with the gas ballast valve closed. Alkaline organic media such as methylamine and dimethylamine can also be pumped satisfactorily, but with the gas ballast valve open.

d) **Elementary gases**
Pumping nitrogen and inert gases requires no special measures.

When handling **hydrogen** it is necessary to make note of the hazard of creating an explosive mixture. The gas ballast valve may in no case be opened when dealing with hydrogen. The motors driving the pumps must be of explosion-proof design.

Oxygen: Particular caution is required when pumping pure oxygen!
Specially formulated pump oils must be used for this purpose. We can supply these, accompanied by an approval certificate issued by the German Federal Materials Testing Authority (BAM), following consultation.

e) **Alkanes**
The low molecular weight alkanes such as methane and butane can be pumped with the gas ballast valve closed or using inert gas as the gas ballast and/or at increased temperature of the pump. But important – Increased explosion hazard!

f) **Alcohols**
Once operating temperature has been reached, methanol and ethanol can be extracted without using gas ballast (N 62 pump oil). To pump higher molecular weight alcohols (e.g. butanol) the gas ballast valve will have to be opened or other protective measures will have to be implemented to prevent condensation.

g) Solvents
Acetone: Can be extracted without difficulty; wait until normal operating temperature is reached.

Benzene: Caution – vapors are highly flammable. Ultimate pressure is degraded by dilution of the pump oil. Mixtures of air and benzene are explosive and flammable. Work without a gas ballast! Inert gases may possibly be used as ballast gas.

Carbon tetrachloride and trichlorethylene: Amenable to pumping; non-flammable and non-explosive but will be dissolved in oil and thus increase the ultimate pressure; wait until normal operating temperature is achieved.

Keep the gas ballast open when pumping carbon tet and other non-flammable solvents. Use N 62 pump oil.

Toluene: Pump through the low-temperature baffle and without gas ballast. Use inert gas, not air, as the gas ballast.

8.3.1.4 Operating defects while pumping with gas ballast – Potential sources of error where the required ultimate pressure is not achieved

a) The pump oil is contaminated (particularly with dissolved vapors). Check the color and properties. Remedy: Allow the pump to run for an extended period of time with the vacuum vessel isolated and the gas ballast valve open. In case of heavy contamination an oil change is advisable. The pump should never be allowed to stand for a longer period of time when it contains contaminated oil.

b) The sliding components in the pump are worn or damaged. Under clean conditions our pumps can run for many years without any particular mainte-nance effort. Where the pump has been allowed to run for a longer period of time with dirty oil, however, the bearings and the gate valves may exhibit mechanical damage. This must always be assumed when the pump no longer achieves the ultimate pressure specified in the catalog even though the oil has been changed. In this case the pump should be sent in for repair or our customer service department should be contacted.

c) The measurement instrument is soiled (see Section 8.4.2).

Potential sources of error when the pump no longer turns

- Check the pump electrical supply.
- The pump has stood for a long time containing contaminated or resinous oil.
- The pump is colder than 10°C and the oil is stiff. Heat the pump.
- There is a mechanical error. Please contact our customer service department.

Oil exits at the shaft

If oil is discharged at the shaft, then the Seeger rotary shaft circlip in the drive bearing will have to be inspected and possibly replaced. The design of the pumps makes it possible to replace this ring easily, following the operating instructions provided with the unit.

8.3.2 Roots pumps

8.3.2.1 General operating instructions, installation and commissioning

Roots pumps must be exactly level. When attaching the pump it is necessary to ensure that the pump is not under any tension or strain whatsoever. Any strains in the pump casing caused by the connection lines shall **be avoided**. Any strain to which the pump is subjected will endanger the pump since the clearances inside the roots pump are very small.

Roots pumps are connected to the line power supply via the motor terminalstrip; a motor protection switch (overload/ overheating) shall be provided as required by local codes.

The direction of motor rotation shall be checked with the intake and outlet ports open prior to installing the pump. The drive shaft, seen from the motor end, must rotate counter-clockwise. Note the arrow on the motor indicating the direction of rotation! If the roots pump runs in the wrong direction, then it is reversed by interchanging two of the phases at the motor connection cord.

The roots pump may be switched on only after the roughing pump has evacuated the vacuum vessel down to the cut-in pressure.

Permissible cut-in pressure P_E will depend on the reduction ratios of the roots pump as against the roughing pump and is calculated by dividing the permissible pressure differential Δp_{max} by the compression ratio, reduced by the value of 1:

$$p_E = \frac{\Delta p_{max}}{k_{th} - 1} \quad \text{where}$$

$$k_{th} = \frac{\text{Theoretical pumping speed for the roots pump}}{\text{Nominal pumping speed for the roughing pump}}$$

If the pump is protected using a diaphragm-type pressure switch, then the pump will be switched on automatically. If a combination of roots pump and roughing pump is to convey highly volatile substances such as liquids with a low boiling point, then it is advisable to use a roots pump which is equipped with an integral bypass line and a valve which will respond to a pre-set pressure. Example: Roots vacuum pumps RUVAC WAU / WSU.

Roots pumps from the RUVAC-WAU/WSU series, being equipped with bypass lines, can generally be switched on together with the forepump. The bypass protects these roots pumps against overloading.

8.3.2.2 Oil change, maintenance work

Under clean operating conditions the oil in the roots pump will be loaded only as a result of the natural wear in the bearings and in the gearing. We nevertheless recommend making the first oil change after about 500 hours in service in order to remove any metal particles which might have been created by abrasion during the run-in period. Otherwise it will be sufficient to change the oil every 3000 hours in operation. When extracting gases containing dust or where other contaminants are present, it will be necessary to change the oil more frequently. If the pumps have to run at high ambient temperatures, then the oil in the sealing ring chamber shall also be replaced at each oil change.

We recommend using our specially formulated N 62 oil.

Under “dirty” operating conditions it is possible for dust deposits to form a “crust” in the pump chamber. These contaminants will deposit in part in the pumping chamber and in part on the pump’s impellers. They may be removed, once the two connection lines have been detached, either by blowing out the system with dry compressed air or by rinsing with a suitable cleaning agent, such as petroleum ether (naphtha).

The oil in the roots pump will then have to be changed. The rotor may be turned only by hand during cleaning since, due to the high speed when the motor is running, the deposits could damage the pump as they dislodge from the surfaces.

Grime which cannot be eliminated by washing can be removed mechanically using a wire brush, metallic scrubber or scraper.

Important!
The dislodged residues may not remain in the pump chamber. After cleaning is completed check the pump for operability by slowly turning the impellers by hand. There may be no resistance to rotation. It is generally not necessary to dismantle the roots pump. If this should nevertheless be required due to heavy soiling, then it is highly advisable to have this done by the manufacturer.

8.3.2.3 Actions in case of operational disturbances

1. **Pump becomes too warm:** (maximum operating temperature 100 to 115 °C)

Possible causes:

- **Overloading:** Excessive heat of compression due to an excessively high pressure ratio. Check the pressure value adjustments and the tightness of the vacuum chamber!
- **Incorrect clearances:** The distances between the rotors and the housings have been narrowed due to dirt or mechanical strain.
- **Soiled bearings:** Excessive friction in the bearings
- **Improper oil level:** If the oil level is too high, the gears will touch the oil, causing friction resistance. Where the oil level is too low the system will not be lubricated.
- **Incorrect oil type:** An SAE 30 grade oil must be used for the pump.

2. **Excessive power consumption:** All the factors which can lead to elevated temperatures can also cause excessisve amounts of power to be drawn. The motor is defective if excessive power requirements are not accompanied by a rise in the temperature at the pump.

3. **Oiling at the pump chamber:**

Possible causes:

- **Oil level too high:** Oil is subjected to excessive thermal loading. Oil foam is swept along.
- **Oil mixed with the product:** Azeotropic degasification of the oil.
- **Pump leaking:** Air ingress through the oil drain or filler screw will cause a large stream of air and conveyance of oil into the pumping chamber.

4. **Abnormal running noises:**

Possible causes:

- **Grime at the impeller**
- **Bearing or gearing damage**
- **Impellers are touching the housing**

In the case of bearing or gearing damage or where the impeller scrapes the housing the pump should be repaired only by the manufacturer.

8.3.3 Turbomolecular pumps

8.3.3.1 General operating instructions

In spite of the relatively large gap between the pump rotor and the stator, the turbomolecular pumps should be protected against foreign objects entering through the intake port. It is for this reason that the pump should never be operated without the supplied wire-mesh splinter guard. In addition, sharp shock to the pump when running and sudden changes in attitude are to be avoided.

Over and above this, and particularly for the larger types with long rotor blades, airing the pump to atmospheric pressure while the impellers are rotating may be done only when observing exactly the rules given in the operating instructions. Under certain circumstances it is possible to operate turbomolecular pumps under exceptional conditions.

The turbomolecular pump may, for example, be used unprotected inside magnetic fields if the magnetic induction at the surface of the pump casing does not exceed $B = 3 \cdot 10^{-3}$ T when radially impinging and $B = 15 \cdot 10^{-3}$ T at axial impingement.

In a radioactive environment standard turbomolecular pumps can be used without hazard at dose rates of from 10^4 to 10^5 rad. If higher dose rates are encountered, then certain materials in the pump can be modified in order to withstand the greater loading. The electronic frequency converters in such cases are to be set up outside the radioactive areas since the semiconductors used inside them can tolerate a dose rate of only about 10^3 rad. The use of motor-driven frequency converters which can withstand up to 10^8 rad represents another option.

Roughing (backing) pumps are required for the operation of turbomolecular pumps. Depending on the size of the vessel to be evacuated, the turbomolecular pumps and forepumps may be switched on simultaneously. If the time required to pump the vessel down to about 1 mbar using the particular fore-pump is longer than the run-up time for the pump (see operating instructions), then it is advisable to delay switching on the turbomolecular pump. Bypass lines are advisable when using turbomolecular pumps in systems set up for batch (cyclical) operations in order to save the run-up time for the pump. Opening the high vacuum valve is not dangerous at pressures of about 10^{-1} mbar.

8.3.3.2 Maintenance

Turbomolecular pumps and frequency converters are nearly maintenance-free. In the case of oil-lubricated pumps it is necessary to replace the bearing lubricant at certain intervals (between 1500 and 2500 hours in operation, depending on the type). This is not required in the case of grease-lubricated pumps (lifetime lubrication). If it should become necessary to clean the pump's turbine unit, then this can easily be done by the customer, observing the procedures described in the operating instructions.

8.3.4 Diffusion and vapor-jet vacuum pumps

8.3.4.1 Changing the pump fluid and cleaning the pump

Changing the pump fluid is always necessary whenever the pump no longer achieves the required ultimate vacuum or when its pumping speed falls off. The service life of the pump fluid will as a rule come to several weeks or months – or even years – and will depend largely on the operating conditions for the pump. It is reduced by frequent pumping at high pressures, by exposure to aggressive vapors and by air ingress of longer duration (this does not apply to silicone oil and mercury).

In the case of oil diffusion and vapor-jet pumps the danger presented to the pump fluid where there is air ingress with the pump hot is often overestimated. Where air ingress (up to atmospheric pressure) is encountered only occasionally and for short periods of time then silicone oil will not be attacked at all and the DIFFELEN pump fluid will only barely be affected. The products with considerably higher vapor pressures which can be created through oxidation are removed again in a short period of time by the fractionation and degassing equipment in the pump (see Section 2.1.6.1). Even though the pump fluid which was originally light in color has been turned brown by air ingress, this need not necessarily mean that the medium has become unusable. If, on the other hand, the pump fluid has turned cloudy and has become more viscous, as well (which may be the case following periods of air ingress lasting for several minutes) then the medium will have to be replaced. It is possible that under certain circumstances cracking products from the pump fluid may make the oil in the forepump unserviceable so that here, too, an oil change will have to be made.

Mercury diffusion and vapor-jet pumps are less sensitive to air ingress than oil diffusion pumps. The oxidation of the hot mercury caused by the air ingress is negligible in regard to the operating characteristics of the pump when compared with the mercury loss in the forepump line.

Changing the pump fluid: The interior section will be extracted from the pump and the contaminated pump fluid poured out. The interior section and the pump body are then cleaned with pure petroleum ether (naphtha). The interior section and pump body of mercury pumps should have been cleaned beforehand with a clean brush; use a bottle brush for the nozzle bores. Ensure that all the nozzle orifices are properly cleaned. It is advantageous to evaporate all solvent residues in a drying kiln. Then the inside section is inserted once again and the fresh pump fluid is installed through the forevacuum port. It is necessary to ensure that the upper nozzle cover is not moistened with pump fluid! Do not install too much pump fluid!

8.3.4.2 Operating errors with diffusion and vapor-jet pumps

Potential sources of defects when the desired ultimate pressure is not reached

- Coolant temperature is too high; inadequate water throughput. The coolant flow should always be monitored by a water flowmeter in order to protect the pump from damage. Remedy: Measure the exit temperature of the coolant water (it should not exceed 30 °C). Increase the coolant flow-through rate. The cooling coils at the pump may have to be decalcified.
- Forevacuum pressure too high: This is possible particularly where vapors which are either evacuated from the vessel or are created as cracking products from the driving medium (e.g. following air ingress) get into the roughing pump. Check the forevacuum pressure with the oil diffusion pump disconnected. Remedy: Run the forevacuum pump for an extended period of time with gas ballast. If this is not sufficient, then the oil in the forepump will have to be changed.
- Pump fluid at the diffusion pump spent or unserviceable: Replace the driving medium.
- Heating is incorrect: Check the heating output and check for good thermal contact between the heating plate and the bottom of the boiler section. Replace the heating unit if necessary.
- Leaks, contamination:
  Remedy: if the pump has been contaminated by vapors it may help to use a metering valve to pass air through the apparatus for some period of time; here the pressure should not exceed 10^{-2} mbar when DIFFELEN is being used.
- Measurement system old or soiled (see Section 8.4.2).

Potential sources of error where there is insufficient pumping speed:

- Forevacuum pressure is too high: Check the forevacuum; allow the gas ballast pump to run for a longer period of time with the gas ballast valve open. It may be necessary to change the oil in the forepump.
- The pump fluid in the diffusion pump has become unserviceable: Driving medium will have to be replaced.
- Nozzles at the diffusion pump are clogged: Clean the diffusion pump.
- Heating is too weak: Check heating output; examine heating plate for good thermal contact with the bottom of the boiler chamber.
- Substances are present in the vacuum vessel which have a higher vapor pressure than the driving medium being used: among these are, for example, mercury, which is particularly hazardous because the mercury vapors will form amalgams with the nonferrous metals in the oil diffusion pump and thus make it impossible to achieve perfect vacuums.

8.3.5 Adsorption pumps

8.3.5.1 Reduction of adsorption capacity

A considerable reduction in pumping speed and failure to reach the ultimate pressure which is normally attainable in spite of thermal regeneration having been carried out indicates that the zeolite being used has become contaminated by outside substances. It does not make good sense to attempt to rejuvenate the contaminated zeolite with special thermal processes. The zeolite should simply be replaced.

8.3.5.2 Changing the molecular sieve

It will be necessary to rinse the adsorption pump thoroughly with solvents before installing the new zeolite charge. Before putting the adsorption pump which has been charged with fresh zeolite into service it is also necessary to bake out the new zeolite charge, under vacuum and using the heating

element associated with the pump, for a period of several hours so that contaminants which might have collected during the storage period can dissipate.

8.3.6 Titanium sublimation pumps

Each of the turns in the titanium sublimation pump contains approximately 1.2 g of useable titanium supply. At a heating current of 50 A the surface temperature comes to about 1850 K, the sublimation rate approximately 0.12 g/h, i.e. a turn can be operated continuously for about 10 hours. Since at pressures below $1 \cdot 10^{-6}$ mbar sublimation is not continuous but rather only at intervals which – at low pressures (below $5 \cdot 10^{-8}$ mbar) and low gas volumes – are already more than ten times the actual sublimation period, one may assume a pumping period of almost one month at a working pressure of 10^{-10} mbar per turn.

The effective pumping speed of a titanium sublimation pump will depend on the getter screen surface and the geometry of the suction opening. The pumping speed, referenced to the surface area of the getter surface, will be dependent on the type of gas and the getter screen temperature. Since inert gases, for example, cannot be pumped at all, titanium sublimation pumps should always be used only with an auxiliary pump (sputter-ion pump, turbomolecular pump) used to pump these gas components. The supplementary pump can be much smaller than the titanium sublimation pump. Only in a few special cases can one do without the additional pump.

The selection of the coolant is dictated by the working conditions and the requirements in terms of ultimate pressure. At high pressures, above $1 \cdot 10^{-6}$ mbar, more thermal energy is applied to the getter screen by frequent sublimation cycles. It is for this reason that cooling with liquid nitrogen is more favorable. At low pressures water cooling may be sufficient. The getter screen should if at all possible be heated to room temperature before airing the pump as otherwise the humidity in the air would condense out on the surface.

8.3.7 Sputter-ion pumps

Sputter-ion pumps use high-voltage current. Installation and connection should be carried out only under the supervision of a qualified specialist. The operating instructions shall be observed!

The service life of sputter-ion pumps depends linearly on the pump's operating pressure. In the case of pumps manufactured by LEYBOLD, the following applies:

$$p \cdot T = 45 \cdot 10^{-3} \text{ mbar} \cdot h$$

(p = operating pressure, T = service life)

This means that for operating pressure of

10^{-3} mbar the service life is 45	hours
10^{-6} mbar the service life is 45,000	hours
10^{-9} mbar the service life is 45,000,000	hours

If a triode pump is not needed over an extended period of time it can either be operated continuously at low pressure – with practically no influence on the service life – or it can be aired, removed from the pump and packed in a dust-tight container. The starting properties of the sputter-ion (triode) pumps manufactured by LEYBOLD are so good that no problems will be encountered when returning the units to service, even after a longer period in storage.

When the sputter-ion pumps are installed one should ensure that the magnetic fields will not interfere with the operation of other devices (ionization vacuum gauges, partial pressure measurement units, etc.). Mounting devices for the sputter-ion pumps may not short circuit the inductance flow and thus weaken the air gap inductance and pumping speed.

If the ultimate pressure which can be attained is not satisfactory even though the apparatus is properly sealed, then it will usually be sufficient to bake out the attached equipment at about 200 to 250 °C. If the pressure here rises to about $1 \cdot 10^{-5}$ mbar when this is done, then the sputter ion pump will become so hot after evacuating the gases for two hours that it will not be necessary to heat it any further in addition. It is also possible to heat the pump by allowing air to enter for 2 hours at 10^{-5} mbar before the other apparatus is then subsequently baked out. If the ultimate pressure is still not satisfactory, then the pump itself will have to be baked out for a few hours at 250 to 300°C (but not higher than 350 °C!). The pump should without fail remain in operation throughout this period! If the pressure rises above $5 \cdot 10^{-5}$ mbar it will be necessary either to heat more slowly or to connect an auxiliary pump. Before airing one should allow the hot sputter-ion pump enough time to cool down at least to 150 °C.

8.4 Information on working with vacuum gauges

8.4.1 Information on installing vacuum sensors

Here both the external situation in the immediate vicinity of the vacuum apparatus and the operating conditions within the apparatus (e.g. working pressure, composition of the gas content) will be important. It is initially necessary to ascertain whether the measurement system being installed is sensitive in regard to its physical attitude. Sensors should only be installed vertically with the vacuum flange at the bottom to keep condensates, metal flakes and filings from collecting in the sensor or even small components such as tiny screws and the like from falling into the sensorand the measurement system. The hot incandescent filaments could also bend and deform improperly and cause electrical shorts inside the measurement system. This is the reason behind the following general rule: **If at all possible, install sensors vertically and open to the bottom.** It is also very important to install measurement systems if at all possible at those points in the vacuum system which will remain free of vibration during operation.

The outside temperature must be taken into account and above all it is necessary to avoid hot kilns, furnaces or stoves or other sources of intense radiation which generate an ambient temperature around the measurement system which lies above the specific acceptable value. Excessive ambient temperatures will result in false pressure indications in thermal conductivity vacuum sensors.

8.4.2 Contamination at the measurement system and its removal

The vacuum gauges used in vacuum technology for pressure measurement will certainly work under “dirty” conditions. This is quite understandable since a vacuum device or system does not serve simply to produce low pressures but rather and primarily have to run processes in chemistry, metallurgy or nuclear physics at low pressures. Here, depending on the nature of the process, considerable quantities of gases or vapors will be liberated either continuously or intermittently; these can pass into the measurement systems provided for pressure measurement and installed in the vacuum system and – due to surface reactions or through simple deposits – can falsify the pressure measurements considerably. This is true for all types of vacuum gauges whereby, of course, high-sensitivity, high-accuracy measurement systems are particularly susceptible to soiling resulting from the causes named. One can attempt to protect the measurement systems against contamination by providing suitable shielding. This, however, will often lead to the pressure registered by the measurement system – which is indeed clean – deviating considerably from the pressure actually prevailing in the system.

It is not fundamentally possible to keep the measurement system in a vacuum gauge from becoming soiled. Thus it is necessary to ensure that

- the influence of the contamination on pressure measurement remains as small as possible and that
- the measurement system can readily be cleaned.

These two conditions are not easy to satisfy by most vacuum gauges in practice.

Dirt in a compression vacuum gauge will cause an incorrect and uncontrollable pressure indication. Dirty THERMOVAC sensors will show a pressure which is too high in the lower measurement range since the surface of the hot wire has changed. In Penning vacuum gauges contamination will induce pressure readings which are far too low since the discharge currents will become smaller. In the case of ionization vacuum gauges with hot cathodes, electrodes and the tube walls can be soiled which, under certain circumstances, will result in a reduction of dielectric strengths. Here, however, the measurement systems can usually be baked out and degassed by passing a current through or by electron bombardment, quite aside from the fact that ionization vacuum gauges are often used in the ultrahigh vacuum range where it is necessary to ensure clean operating conditions for other reasons.

8.4.3 The influence of magnetic and electrical fields

In all those measurement instruments which use the ionization of gas molecules as the measurement principle (cold-cathode and hot-cathode ionization vacuum gauges), strong magnetic leakage fields or electrical potentials can have a major influence on the pressure indication. At low pressures it is also possible for wall potentials which deviate from the cathode potential to influence the ion trap current.

In vacuum measurement systems used in the high and ultrahigh regimes it is necessary to ensure in particular that the required high insulation values for the high-voltage electrodes and ion traps also be maintained during operation and sometimes even during bake-out procedures. Insulation defects may occur both in the external feed line and inside the measurement system itself. Insufficient insulation at the trap (detector) lead may allow creep currents – at low pressures – to stimulate overly high pressure value readings. The very low ion trap currents make it necessary for this lead to be particularly well insulated. Inside the measurement sensors, too, creep currents can occur if the trap is not effectively shielded against the other electrodes.

An error frequently made when connecting measurement sensors to the vacuum system is the use of connector piping which is unacceptably long and narrow. The conductance value must in all cases be kept as large as possible. The most favorable solution is to use integrated measurement systems. Whenever connector lines of lower conductance values are used the pressure indication, depending on the cleanliness of the measurement sensors and the connector line, may be either too high or too low. Here measurement errors by more than one complete order of magnitude are possible! Where systems can be baked out it is necessary to ensure that the connector line can also be heated.

8.4.4 Connectors, power pack, measurement systems

The measurement cables (connector cables between the sensor and the vacuum gauge control unit) are normally 2 m long. If longer measurement cables must be used – for installation in control panels, for example – then it will be necessary to examine the situation to determine whether the pressure reading might be falsified. Information on the options for using overlength cables can be obtained from our technical consulting department.

9. Tables, formulas, nomograms and symbols

Unit	N · m^{-2}, Pa [2]	mbar	bar	Torr
1 N · m^{-2} (= 1 Pa)	1	1 · 10^{-2}	1 · 10^{-5}	7.5 · 10^{-3}
1 mbar	100	1	1 · 10^{-3}	0.75
1 bar	1 · 10^{5}	1 · 103	1	750
1 Torr [3]	133	1.33	1.33 · 10^{-3}	1

1) The torr is included in the table only to facilitate the transition from this familiar unit to the statutory units N · m^{-2}, mbar and bar. In future the pressure units torr, mm water column, mm mercury column (mm Hg), % vacuum, technical atmosphere (at), physicalatmosphere (atm), atmosphere absolute (ata), pressure above atmospheric and pressure below atmospheric may no longer be used. Reference is made to DIN 1314 in this context.
2) The unit Newton divided by square meters (N · m^{-2}) is also designated as Pascal (Pa): 1 N · m^{-2}= 1 Pa.
Newton divided by square meters or Pascal is the SI unit for the pressure of fluids.
3) 1 torr = 4/3 mbar; fl torr = 1 mbar.

Table I: Permissible pressure units including the torr 1) and its conversion

Abbrev.	Gas	C* = λ · p [cm · mbar]
H_2	Hydrogen	12.00 · 10^{-3}
He	Helium	18.00 · 10^{-3}
Ne	Neon	12.30 · 10^{-3}
Ar	Argon	6.40 · 10^{-3}
Kr	Krypton	4.80 · 10^{-3}
Xe	Xenon	3.60 · 10^{-3}
Hg	Mercury	3.05 · 10^{-3}
O_2	Oxygen	6.50 · 10^{-3}
N_2	Nitrogen	6.10 · 10^{-3}
HCl	Hydrochloric acid	4.35 · 10^{-3}
CO_2	Carbon dioxide	3.95 · 10^{-3}
H_2O	Water vapor	3.95 · 10^{-3}
NH_3	Ammonia	4.60 · 10^{-3}
C_2H_5OH	Ethanol	2.10 · 10^{-3}
Cl_2	Chlorine	3.05 · 10^{-3}
Air	Air	6.67 · 10^{-3}

Table III: Mean free path l
Values of the product c* of the mean free path λ (and pressure p for various gases at 20 °C (see also Fig. 9.1)

1 ↓ = ... →	mbar	Pa (N/m^3)	dyn · cm^{-2} (µbar)	atm (phys.)	Torr (mm Hg)	inch Hg	Micron (µ)	cm H_2O	kp · cm^{-2} (at tech.)	lb · in^{-2} (psi)	lb · ft^{-2}
mbar	1	10^2	10^3	9.87 · 10^{-4}	0.75	2.953 · 10^{-2}	7.5 · 10^2	1.02	1.02 · 10^{-3}	1.45 · 10^{-2}	2.089
Pa	10^{-2}	1	10	9.87 · 10^{-6}	7.5 · 10^{-3}	2.953 · 10^{-4}	7.5	1.02 · 10^{-2}	1.02 · 10^{-5}	1.45 · 10^{-4}	2.089 · 10^{-2}
µbar	10^{-3}	0.1	1	9.87 · 10^{-7}	7.5 · 10^{-4}	2.953 · 10^{-5}	7.5 · 10^{-1}	1.02 · 10^{-3}	1.02 · 10^{-6}	1.45 · 10^{-5}	2.089 · 10^{-3}
atm	1013	1.01 · 10^5	1.01 · 10^6	1	760	29.92	7.6 · 10^5	1.03 · 10^3	1.033	14.697	2116.4
Torr	1.33	1.33 · 10^2	1.33 · 10^3	1.316 · 10^{-3}	1	3.937 · 10^{-2}	10^3	1.3595	1.36 · 10^{-3}	1.934 · 10^{-2}	2.7847
in Hg	33.86	33.9 · 10^2	33.9 · 10^3	3.342 · 10^{-2}	25.4	1	2.54 · 10^4	34.53	3.453 · 10^{-2}	0.48115	70.731
µ	1.33 · 10^{-3}	1.33 · 10^{-1}	1.333	1.316 · 10^{-6}	10^{-3}	3.937 · 10^{-5}	1	1.36 · 10^{-3}	1.36 · 10^{-6}	1.934 · 10^{-5}	2.785 · 10^{-3}
cm H_2O	0.9807	98.07	980.7	9.678 · 10^{-4}	0.7356	2.896 · 10^{-2}	7.36 · 10^2	1	10^{-3}	1.422 · 10^{-2}	2.0483
at	9.81 · 10^2	9.81 · 10^4	9.81 · 10^5	0.968	7.36 · 10^2	28.96	7.36 · 10^5	103	1	14.22	2048.3
psi	68.95	68.95 · 10^2	68.95 · 10^3	6.804 · 10^{-2}	51.71	2.036	51.71 · 10^3	70.31	7.03 · 10^{-2}	1	1.44 · 10^2
lb · ft^{-2}	0.4788	47.88	478.8	4.725 · 10^{-4}	0. 3591	1.414 · 10^{-2}	359.1	0.488	4.88 · 10^{-4}	6.94 · 10^{-3}	1

Normal conditions: 0 °C and sea level, i.e. p = 1013 mbar = 760 mm Hg = 760 torr = 1 atm
in Hg = inches of mercury; 1 mtorr (millitorr) = 10^{-3} torr = 1 µ (micron … µm Hg column)
Pounds per square inch = lb · in^{-2} = lb / sqin = psi (psig = psi gauge … pressure above atmospheric, pressure gauge reading; psia = psi absolute … absolute pressure)
Pounds per square foot = lb / sqft = lb / ft^2; kgf/sqcm^2 = kg force per square cm = kp / cm^2 = at; analogously also: lbf / squin = psi
1 dyn · cm^{-2} (cgs) = 1 µbar (microbar) = 1 barye; 1 bar = 0.1 Mpa; 1 cm water column (cm water column = g / cm^2 at 4 °C) = 1 Ger (Geryk)
atm … physical atmosphere – at … technical atmosphere; 100 - (x mbar / 10.13) = y % vacuum

Table II: Conversion of pressure units

Tables, Formulas, Diagrams

Variable	General formula	For easy calculation	Value for air at 20°C
Most probable speed of particles c_w	$c_w = \sqrt{\frac{2 \cdot R \cdot T}{M}}$	$c_w = 1.29 \cdot 10^4 \sqrt{\frac{T}{M}} \left[\frac{cm}{s}\right]$	$c_w = 410$ [m/s]
Mean velocity of particles $\bar{c}$	$\bar{c} = \sqrt{\frac{8 \cdot R \cdot T}{\pi \cdot M}}$	$\bar{c} = 1.46 \cdot 10^4 \sqrt{\frac{T}{M}} \left[\frac{cm}{s}\right]$	$\bar{c} = 464$ [m/s]
Mean square of velocity of particles $\bar{c^2}$	$\bar{c^2} = \frac{3 \cdot R \cdot T}{M}$	$\bar{c^2} = 2.49 \cdot 10^8 \frac{T}{M} \left[\frac{cm^2}{s^2}\right]$	$\bar{c^2} = 25.16 \cdot 10^4 \left[\frac{cm^2}{s^2}\right]$
Gas pressure p of particles	$p = n \cdot k \cdot T$ $p = \frac{1}{3} \cdot n \cdot m_T \cdot \bar{c^2}$ $p = \frac{1}{3} \cdot \cdot \bar{c^2}$	$p = 13.80 \cdot 10^{-20} \cdot n \cdot T$ [mbar]	$p = 4.04 \cdot 10^{-17} \cdot n$ [mbar] (applies to all gases)
Number density of particles n	$n = p/kT$	$n = 7.25 \cdot 10^{18} \frac{p}{T}$ [cm^{-3}]	$p = 2.5 \cdot 10^{16} \cdot p$ [cm^{-3}] (applies to all gases)
Area-related impingement Z_A	$Z_A = \frac{1}{4} \cdot n \cdot \bar{c}$ $Z_A = \sqrt{\frac{N_A}{2 \cdot \pi \cdot M \cdot k \cdot T}} \cdot p$	$Z_A = 2.63 \cdot 10^{22} \cdot \frac{p}{\sqrt{M \cdot T}} p$ [$cm^{-2}\ s^{-1}$]	$Z_A = 2.85 \cdot 10^{20} \cdot p$ [$cm^{-2}\ s^{-1}$] (see Fig. 78.2)
Volume collision rate Z_V	$Z_V = \frac{1}{2} n \cdot \bar{c} \cdot \lambda$ $Z_A = \frac{1}{c^*} \sqrt{\frac{2 \cdot N_A}{\pi \cdot M \cdot k \cdot T}} \cdot p^2$	$Z_V = 5.27 \cdot 10^{22} \cdot \frac{p^2}{c^* \cdot \sqrt{M \cdot T}}$ [$cm^{-3}\ s^{-1}$]	$Z_V = 8.6 \cdot 10^{22} \cdot p^2$ [$cm^{-3}\ s^{-1}$] (see Fig. 78.2)
Equation of state of ideal gas	$p \cdot V = \nu \cdot R \cdot T$	$p \cdot V = 83.14 \cdot \nu \cdot T$ [mbar · ℓ]	$p \cdot V = 2.44 \cdot 10^4\ \nu$ [mbar · ℓ] (for all gases)
Area-related mass flow rate q_m, A	$q_{m,A} = Z_A \cdot m_T = \sqrt{\frac{M}{2 \cdot \pi \cdot k \cdot T \cdot N_A}} \cdot p$	$Q_{m,A} = 4.377 \cdot 10^{-2} \sqrt{\frac{M}{T}} p$ [g $cm^{-2}\ s^{-1}$]	$q_{m,A} = 1.38 \cdot 10^{-2} \cdot p\ g$ [$cm^{-2}\ s^{-1}$]

$c^* = \lambda \cdot p$ in cm · mbar (see Tab. III)
k Boltzmann constant in mbar · l · K^{-1}
λ mean free path in cm
M molar mass in g · mol^{-1}
m_T particle mass in g
N_A Avogadro constant in mol^{-1}
n number density of particles in cm^{-3}
ν amount of substance in mol
p gas pressure in mbar
R molar gas constant in mbar · l · mol^{-1} K^{-1}
T thermodynamic temperature in K
V volume in l

Table IV: Compilation of important formulas pertaining to the kinetic theory of gases

Designation, alphabetically	Symbol	Value and unit	Remarks
Atomic mass unit	m_u	$1.6605 \cdot 10^{-27}$ kg	
Avogadro constant	N_A	$6.0225 \cdot 10^{23}\ mol^{-1}$	Number of particles per mol, formerly: Loschmidt number
Boltzmann constant	k	$1.3805 \cdot 10^{-23}$ J · K^{-1}	
		$13.805 \cdot 10^{-23} \frac{mbar \cdot l}{K}$	
	Electron rest mass	m_e	$9.1091 \cdot 10^{-31}$ kg
	Elementary charge	e	$1.6021 \cdot 10^{-19}$ A · s
Molar gas constant	R	8.314 J · mol^{-1} K^{-1}	
		$= 83.14 \frac{mbar \cdot l}{mol \cdot K}$	$R = N_A \cdot k$
Molar volume of		$22.414\ m^3\ kmol^{-1}$	DIN 1343; formerly: molar volume
the ideal gas	V_o	22.414 l · mol^{-1}	at 0 °C and 1013 mbar
Standard acceleration of free fall	g_n	9.8066 m · s^{-2}	
Planck constant	h	$6.6256 \cdot 10^{-34}$ J · s	
Stefan-Boltzmann constant	σ	$5.669 \cdot 10^{-8} \frac{W}{m^2 K^4}$	also: unit conductance, radiation constant
Specific electron charge	$\frac{-e}{m_e}$	$-1.7588 \cdot 10^{11} \frac{A \cdot s}{kg}$	
Speed of light in vacuum	c	$2.9979 \cdot 10^8$ m · s^{-1}	
Standard reference density of a gas	ϱ_n	kg · m^{-3}	Density at $\vartheta = 0$ °C and $p_n = 1013$ mbar
Standard reference pressure	p_n	101.325 Pa = 1013 mbar	DIN 1343 (Nov. 75)
Standard reference temperature	T_n	$T_n = 273.15$ K, $\vartheta = 0$ °C	DIN 1343 (Nov. 75)

Table V: Important values

Tables, Formulas, Diagrams

Unit	$l \cdot s^{-1}$	$m^3 \cdot h^{-1}$	$cm^3 \cdot s^{-1}$	$cuft \cdot min^{-1}$
$1\ l \cdot s^{-1}$	1	3.6	1000	2.12
$1\ m^3 \cdot h^{-1}$	0.2778	1	277.8	0.589
$1\ cm^3 \cdot s^{-1}$	10^{-3}	$3.6 \cdot 10^{-3}$	1	$2.1 \cdot 10^{-3}$
$1\ cuft \cdot min^{-1}$	0.4719	1.699	471.95	1

Table VI: Conversion of pumping speed (volume flow rate) units

$1\downarrow = ... \rightarrow$	mbar · l/s	kg · h⁻¹ (20°C)	kg · h⁻¹ (0°C)	cm³/h (NTP)	cm³/s (NTP)	Torr · l/s	g/a (F12. 20 °C)	g/a (F12. 25 °C)	µ · cfm	lusec	Pa · l/s	slpm
mbar · l/s	1	$4.28 \cdot 10^{-3}$	$4.59 \cdot 10^{-3}$	3554	0.987	0.75	1.56 · 105	1.54 · 105	1593	$7.52 \cdot 10^{2}$	100	$59.2 \cdot 10^{-3}$
kg · h⁻¹ (20 °C)	234	1	1.073	$8.31 \cdot 10^{5}$	231	175	–	–	$37.2 \cdot 10^{4}$	$1.75 \cdot 10^{5}$	$23.4 \cdot 10^{3}$	13.86
kg · h⁻¹ (0 °C)	218	0.932	1	$7.74 \cdot 10^{5}$	215	163	–	–	$34.6 \cdot 10^{4}$	$1.63 \cdot 10^{5}$	$21.8 \cdot 10^{3}$	12.91
cm³/h (NTP)	$2.81 \cdot 10^{-4}$	$1.20 \cdot 10^{-6}$	$1.29 \cdot 10^{-6}$	1	$2.78 \cdot 10^{-4}$	$2.11 \cdot 10^{-4}$	44	–	$44.7 \cdot 10^{-2}$	$2.11 \cdot 10^{-1}$	$2.81 \cdot 10^{-2}$	$1.66 \cdot 10^{-5}$
cm³/s (NTP)	1.013	$4.33 \cdot 10^{-3}$	$4.65 \cdot 10^{-3}$	3600	1	0.760	$1.58 \cdot 10^{5}$	–	1611	760	101	$6 \cdot 10^{-2}$
Torr · l/s	1.33	$5.70 \cdot 10^{-3}$	$6.12 \cdot 10^{-3}$	4727	1.32	1	$2.08 \cdot 10^{5}$	$2.05 \cdot 10^{5}$	2119	$1 \cdot 10^{3}$	133	$78.8 \cdot 10^{-3}$
g/a (F12. 20 °C)	$6.39 \cdot 10^{-6}$	–	–	$2.27 \cdot 10^{-2}$	$6.31 \cdot 10^{-6}$	$4.80 \cdot 10^{-6}$	1	–	$10.2 \cdot 10^{-3}$	$4.8 \cdot 10^{-3}$	$6.39 \cdot 10^{-4}$	$37.9 \cdot 10^{-8}$
g/a (F12. 25 °C)	$6.50 \cdot 10^{-6}$	–	–	–	–	$4.88 \cdot 10^{-6}$	–	1	$10.4 \cdot 10^{-3}$	$4.89 \cdot 10^{-3}$	$6.5 \cdot 10^{-4}$	$38.5 \cdot 10^{-8}$
µ · cfm	$6.28 \cdot 10^{-4}$	$2.69 \cdot 10^{-6}$	$2.89 \cdot 10^{-6}$	2.24	$6.21 \cdot 10^{-4}$	$4.72 \cdot 10^{-4}$	98.16	96.58	1	0.472	$6.28 \cdot 10^{-2}$	$37.2 \cdot 10^{-6}$
lusec	$1.33 \cdot 10^{-3}$	$5.70 \cdot 10^{-6}$	$6.12 \cdot 10^{-6}$	4.737	$1.32 \cdot 10^{-3}$	$1 \cdot 10^{-3}$	208	205	2.12	1	$13.3 \cdot 10^{-2}$	$78.8 \cdot 10^{-6}$
Pa · l/s	$1 \cdot 10^{-2}$	$4.28 \cdot 10^{-5}$	$4.59 \cdot 10^{-5}$	35.54	$9.87 \cdot 10^{-3}$	$7.5 \cdot 10^{-3}$	$1.56 \cdot 10^{3}$	$1.54 \cdot 10^{3}$	15.93	7.50	1	$59.2 \cdot 10^{-5}$
slpm	16.88	$72.15 \cdot 10^{-3}$	$77.45 \cdot 10^{-3}$	$60.08 \cdot 10^{3}$	16.67	12.69	$2.64 \cdot 10^{6}$	$2.60 \cdot 10^{6}$	$26.9 \cdot 10^{3}$	$12.7 \cdot 10^{3}$	$16.9 \cdot 10^{2}$	1

1 cm³ (NTP) = 1 cm³ under normal conditions (T = 273.15 K; p = 1013.25 mbar)

NTP = normal temperature and pressure (1 atm; 0 °C) R = 83.14 mbar · l · mol⁻¹ · K⁻¹

1 cm³ (NTP) · h⁻¹ = 1 atm · cm³ · h⁻¹ = 1 Ncm³ · h⁻¹ = 1 std cch

SI coherent: 1 Pa · m³ · s⁻¹ = 10 mbar · l · s⁻¹; R = 8.314 Pa · m³ · mol⁻¹ · K⁻¹; M in kg / mol

1 cm³ (NTP) · s⁻¹ = 1 sccs = 60 cm³ (NTP) · min⁻¹ 60 sccm = 60 std ccm = 60 Ncm³ · min⁻¹

1 lusec = 1 l · µ · s⁻¹ 1 · µ = 1 micron = 10^{-3} Torr 1 lusec = 10^{-3} Torr · l · s⁻¹

Freon F 12 (CCl_2F_2) M = 120.92 g · mol⁻¹; air M = 28.96 g · mol⁻¹

1 sccm = 10^{-3} slpm = 10^{-3} N · l · min⁻¹ = 60 sccs

Note: Anglo-American units are not abbreviated nonuniformly! Example: Standard cubic centimeter per minute → sccm = sccpm = std ccm = std ccpm

Table VIIa: Conversion of throughput (Q_{pV}) units; (leak rate) units

$1\downarrow = ... \rightarrow$	mbar · l/s	cm³/s **)	Torr · l/s	Pa · m³/s	g/a *)	oz/yr *)	lb/yr *)	atm · ft³/min	µ · l/s	µ · ft³/h	µ · ft³/min
mbar · l/s	1	0.987	0.75	10^{-1}	$1.56 \cdot 10^{5}$	$5.5 \cdot 10^{3}$	$3.4 \cdot 10^{2}$	$2.10 \cdot 10^{-3}$	$7.52 \cdot 10^{2}$	$9.56 \cdot 10^{4}$	1593
cm³/s **)	1.013	1	0.76	$1.01 \cdot 10^{-1}$	$1.58 \cdot 10^{5}$	$5.6 \cdot 10^{3}$	$3.44 \cdot 10^{2}$	$2.12 \cdot 10^{-3}$	760	$96.6 \cdot 10^{3}$	1614
Torr · l/s	1.33	1.32	1	$1.33 \cdot 10^{-1}$	$2.08 \cdot 10^{5}$	$7.3 \cdot 10^{3}$	$4.52 \cdot 10^{2}$	$2.79 \cdot 10^{-3}$	10^{3}	$1.27 \cdot 10^{5}$	2119
Pa · m³/s	10	9.9	7.5	1	$1.56 \cdot 10^{6}$	$5.51 \cdot 10^{4}$	$3.4 \cdot 10^{3}$	$2.09 \cdot 10^{-2}$	$7.5 \cdot 10^{3}$	$9.54 \cdot 10^{5}$	$15.9 \cdot 10^{3}$
g/a *)	$6.39 \cdot 10^{-6}$	$6.31 \cdot 10^{-6}$	$4.80 \cdot 10^{-6}$	$6.41 \cdot 10^{-7}$	1	$3.5 \cdot 10^{-2}$	$2.17 \cdot 10^{-3}$	$1.34 \cdot 10^{-8}$	$4.8 \cdot 10^{-3}$	0.612	$10.2 \cdot 10^{-3}$
oz/yr *)	$1.82 \cdot 10^{-4}$	$1.79 \cdot 10^{-4}$	$1.36 \cdot 10^{-4}$	$1.82 \cdot 10^{-5}$	28.33	1	6.18 ·· 10^{-2}	$3.80 \cdot 10^{-7}$	0.136	17.34	0.289
lb/yr *)	$2.94 \cdot 10^{-3}$	$2.86 \cdot 10^{-3}$	$2.17 \cdot 10^{-3}$	$2.94 \cdot 10^{-4}$	$4.57 \cdot 10^{2}$	16	1	$6.17 \cdot 10^{-6}$	2.18	280	4.68
atm · ft³/min	$4.77 \cdot 10^{2}$	$4.72 \cdot 10^{2}$	$3.58 \cdot 10^{2}$	47.7	$7.46 \cdot 10^{7}$	$2.63 \cdot 10^{6}$	$1.62 \cdot 10^{5}$	1	$3.58 \cdot 10^{5}$	$4.55 \cdot 10^{7}$	$7.60 \cdot 10^{5}$
µ · l/s	$1.33 \cdot 10^{-3}$	$1.32 \cdot 10^{-3}$	10^{-3}	$1.33 \cdot 10^{-4}$	208	7.34	$4.52 \cdot 10^{-1}$	$2.79 \cdot 10^{-6}$	1	127	2.12
µ · ft³/h	$1.05 \cdot 10^{-5}$	$1.04 \cdot 10^{-5}$	$7.87 \cdot 10^{-6}$	$1.05 \cdot 10^{-6}$	1.63	$5.77 \cdot 10^{-2}$	$3.57 \cdot 10^{-3}$	$2.20 \cdot 10^{-8}$	$7.86 \cdot 10^{-3}$	1	$1.67 \cdot 10^{-2}$
µ · ft³/min	$6.28 \cdot 10^{-4}$	$6.20 \cdot 10^{-4}$	$4.72 \cdot 10^{-4}$	$6.28 \cdot 10^{-5}$	98	3.46	$2.14 \cdot 10^{-1}$	$1.32 \cdot 10^{-6}$	0.472	60	1

1 · µ · ft³ · h⁻¹ = 1.04 · 10^{-5} stsd cc per second

1 micron cubic foot per hour = 0.0079 micron liter per second

1 kg = 2.2046 pounds (lb)

1 cm³ · s⁻¹ (NTP) = 1 atm · cm³ · s⁻¹ = 1 scc · s⁻¹ = 1 sccss

1 micron liter per second = 0.0013 std cc per second = 1 lusec

1 cubic foot (cfut. cf) = 28.3168 dm³

1 atm · ft³ · min⁻¹ = 1 cfm (NTP)

1 micron cubic foot per minute = 1 µ · ft³ · min⁻¹ = 1 µ · cuft · min⁻¹ = 1µ · cfm

1 lb = 16 ounces (oz)

1 Pa · m³/s = 1 Pa · m³/s (anglo-am.) = 10^{3} Pa · l/s

1 standard cc per second = 96.600 micron cubic feet per hour

1 lusec = 1 µ · l · s⁻¹

1 µ · l · s⁻¹ = 127 µ · ft³ · h⁻¹ = 0.0013 std cc per second = 1 lusec

1 std cc/sec = 760 µ · l · s⁻¹

*) F12 (20 °C) $C.Cl_2F_2$ M = 120.92 h/mol

**) (NTP) normal temperature and pressure 1 atm und 0 °C

Table VII b: Conversion of throughput (QpV) units; (leak rate) units

Tables, Formulas, Diagrams

	% by weight	% by volume	Partial pressure mbar
N_2	75.51	78.1	792
O_2	23.01	20.93	212
Ar	1.29	0.93	9.47
CO_2	0.04	0.03	0.31
Ne	$1.2 \cdot 10^{-3}$	$1.8 \cdot 10^{-3}$	$1.9 \cdot 10^{-2}$
He	$7 \cdot 10^{-5}$	$7 \cdot 10^{-5}$	$5.3 \cdot 10^{-3}$
CH_4	$2 \cdot 10^{-4}$	$2 \cdot 10^{-4}$	$2 \cdot 10^{-3}$
Kr	$3 \cdot 10^{-4}$	$1.1 \cdot 10^{-4}$	$1.1 \cdot 10^{-3}$
N_2O	$6 \cdot 10^{-5}$	$5 \cdot 10^{-5}$	$5 \cdot 10^{-4}$
H_2	$5 \cdot 10^{-6}$	$5 \cdot 10^{-5}$	$5 \cdot 10^{-4}$
Xe	$4 \cdot 10^{-5}$	$8.7 \cdot 10^{-6}$	$9 \cdot 10^{-5}$
O_3	$9 \cdot 10^{-6}$	$7 \cdot 10^{-6}$	$7 \cdot 10^{-5}$
	Σ 100 %	Σ 100 %	Σ 1013
50 % RH at 20 °C	1.6	1.15	11.7

Note: In the composition of atmospheric air the relative humidity (RH) is indicated separately along with the temperature. At the given relative humidity, therefore, the air pressure read on the barometer is 1024 mbar.

Table VIII: Composition of atmospheric air

		Rough vacuum	Medium vacuum	High vacuum	Ultrahigh vacuum
Pressure	p [mbar]	1013 – 1	$1 - 10^{-3}$	$10^{-3} - 10^{-7}$	$< 10^{-7}$
Particle number density	n [cm^{-3}]	$10^{19} - 10^{16}$	$10^{16} - 10^{13}$	$10^{13} - 10^{9}$	$< 10^{9}$
Mean free path	λ [cm]	$< 10^{-2}$	$10^{-2} - 10$	$10 - 10^{5}$	$> 10^{5}$
Impingement rate	Z_a [$cm^{-2} \cdot s^{-1}$]	$10^{23} - 10^{20}$	$10^{20} - 10^{17}$	$10^{17} - 10^{13}$	$< 10^{13}$
Vol.-related collision rate	Z_V [$cm^{-3} \cdot s^{-1}$]	$10^{29} - 10^{23}$	$10^{23} - 10^{17}$	$10^{17} - 10^{9}$	$< 10^{9}$
Monolayer time	τ [s]	$< 10^{-5}$	$10^{-5} - 10^{-2}$	$10^{-2} - 100$	> 100
Type of gas flow		Viscous flow	Knudsen flow	Molecular flow	Molecular flow
Other special features		Convection dependent on pressure	Significant change in thermal conductivity of a gas	Significant reduction-in volume related collision rate	Particles on the surfaces dominate to a great extend in relation to particles in gaseous space

Table IX: Pressure ranges used in vacuum technology and their characteristics (numbers rounded off to whole power of ten)

At room temperature									
Standard values[1] ($mbar \cdot l \cdot s^{-1} \cdot cm^{-2}$)			Metals 10^{-9} ... $\cdot 10^{-7}$			Nonmetals 10^{-7} ... $\cdot 10^{-5}$			
Outgassing rates (standard values) as a function of time									
Examples:	1/2 hr.	1 hr.	3 hr.	5 hr.	Examples:	1/2 hr.	1 hr.	3 hr.	5 hr.
Ag	$1.5 \cdot 10^{-8}$	$1.1 \cdot 10^{-8}$	$2 \cdot 10^{-9}$		Silicone	$1.5 \cdot 10^{-5}$	$8 \cdot 10^{-6}$	$3.5 \cdot 10^{-6}$	$1.5 \cdot 10^{-6}$
Al	$2 \cdot 10^{-8}$	$6 \cdot 10^{-9}$			NBR	$4 \cdot 10^{-6}$	$3 \cdot 10^{-6}$	$1.5 \cdot 10^{-6}$	$1 \cdot 10^{-6}$
Cu	$4 \cdot 10^{-8}$	$2 \cdot 10^{-8}$	$6 \cdot 10^{-9}$	$3.5 \cdot 10^{-9}$	Acrylic glass	$1.5 \cdot 10^{-6}$	$1.2 \cdot 10^{-6}$	$8 \cdot 10^{-7}$	$5 \cdot 10^{-7}$
Stainless steel		$9 \cdot 10^{-8}$	$3.5 \cdot 10^{-8}$	$2.5 \cdot 10^{-8}$	FPM, FKM	$7 \cdot 10^{-7}$	$4 \cdot 10^{-7}$	$2 \cdot 10^{-7}$	$1.5 \cdot 10^{-7}$

[1] All values depend largely on pretreatment!

Table X: Outgassing rate of materials in $mbar \cdot l \cdot s^{-1} \cdot cm^{-2}$

Nominal width (DN)	Internal diam. (mm)	
Series	R5	R10
	10	10
	16	16
		20 21
	25	24
		32 34
	40	41
		50 51
	63	70
		80 83
	100	102
		125 127
	160	153
		200 213
	250	261
		320 318
	400	400
		500 501
	630	651
		800 800
	1000	1000

[1] The nominal internal diameters correspond approximately to the internal diameters of the pipeline components" (DIN 2402 - Feb. 1976). The left-hand column of the nominal internal diameter series is preferred in practice.

Table XI: Nominal internal diameters (DN) and internal diameters of tubes, pipes and apertures with circular cross-section (according to PNEUROP).

Solvent	Relative molecular mass	Density g / cm^3 (20 °C)	Melting point °C	Boiling point °C	Maximum admissible concentration (MAC) cm^3 / m^3
Acetone	58	0.798		56	
Benzene (solution)	78	0.8788	5.49	80.2	25
Petrol (light)		0.68 ... 0.72		> 100	
Carbon tetrachloride	153.8	1.592	– 22.9	76.7	25
Chloroform	119.4	1.48	– 63.5	61	50
Diethyl ether	46	0.7967	–114.5	78	1000
Ethyl alcohol	74	0.713	– 116.4	34.6	400
Hexane	86	0.66	– 93.5	71	500
Isopropanol	60.1	0.785	– 89.5	82.4	400
Methanol	32	0.795	– 97.9	64.7	200 (toxic!)
Methylene chloride	85	1.328		41	
Nitromethane	61	1.138	– 29.2	101.75	100
Petroleum ether	mixture	0.64	–	40 ... 60	
Trichlorethylene („Tri")	131.4	1.47		55	
Water	18.02	0.998	0.00	100.0	–

Table XII: Important data (characteristic figures) for common solvents

Tables, Formulas, Diagrams

t °C	ps mbar	ϱ_D g/m³
– 100	$1.403 \cdot 10^{-5}$	$1.756 \cdot 10^{-5}$
– 99	1.719	2.139
– 98	2.101	2.599
– 97	2.561	3.150
– 96	3.117	3.812
– 95	$3.784 \cdot 10^{-5}$	$4.602 \cdot 10^{-5}$
– 94	4.584	5.544
– 93	5.542	6.665
– 92	6.685	7.996
– 91	8.049	9.574
– 90	$9.672 \cdot 10^{-5}$	$11.44 \cdot 10^{-5}$
– 89	11.60	13.65
– 88	13.88	16.24
– 87	16.58	19.30
– 86	19.77	22.89
– 85	$23.53 \cdot 10^{-5}$	$27.10 \cdot 10^{-5}$
– 84	27.96	32.03
– 83	33.16	37.78
– 82	39.25	44.49
– 81	46.38	52.30
– 80	$0.5473 \cdot 10^{-3}$	$0.6138 \cdot 10^{-3}$
– 79	0.6444	0.7191
– 78	0.7577	0.8413
– 77	0.8894	0.9824
– 76	1.042	1.145
– 75	$1.220 \cdot 10^{-3}$	$1.334 \cdot 10^{-3}$
– 74	1.425	1.550
– 73	1.662	1.799
– 72	1.936	2.085
– 71	2.252	2.414
– 70	$2.615 \cdot 10^{-3}$	$2.789 \cdot 10^{-3}$
– 69	3.032	3.218
– 68	3.511	3.708
– 67	4.060	4.267
– 66	4.688	4.903
– 65	$5.406 \cdot 10^{-3}$	$5.627 \cdot 10^{-3}$
– 64	6.225	6.449
– 63	7.159	7.381
– 62	8.223	8.438
– 61	9.432	9.633
– 60	$10.80 \cdot 10^{-3}$	$10.98 \cdot 10^{-3}$
– 59	12.36	12.51
– 58	14.13	14.23
– 57	16.12	16.16
– 56	18.38	18.34
– 55	$20.92 \cdot 10^{-3}$	$20.78 \cdot 10^{-3}$
– 54	23.80	23.53
– 53	27.03	26.60
– 52	30.67	30.05
– 51	34.76	33.90
– 50	$39.35 \cdot 10^{-3}$	$38.21 \cdot 10^{-3}$
– 49	44.49	43.01
– 48	50.26	48.37
– 47	56.71	54.33
– 46	63.93	60.98
– 45	$71.98 \cdot 10^{-3}$	$68.36 \cdot 10^{-3}$
– 44	80.97	76.56
– 43	90.98	85.65
– 42	102.1	95.70
– 41	$114.5 \cdot 10^{-3}$	$106.9 \cdot 10^{-3}$
– 40	0.1283	0.1192
– 39	0.1436	0.1329
– 38	0.1606	0.1480
– 37	0.1794	0.1646
– 36	0.2002	0.1829
– 35	0.2233	0.2032
– 34	0.2488	0.2254
– 33	0.2769	0.2498
– 32	0.3079	0.2767
– 31	0.3421	0.3061
– 30	0.3798	0.3385
– 29	0.4213	0.3739
– 28	0.4669	0.4127
– 27	0.5170	0.4551
– 26	0.5720	0.5015
– 25	0.6323	0.5521
– 24	0.6985	0.6075
– 23	0.7709	0.6678
– 22	0.8502	0.7336
– 21	0.9370	0.8053
– 20	1.032	0.8835
– 19	1.135	0.9678
– 18	1.248	1.060
– 17	1.371	1.160
– 16	1.506	1.269
– 15	1.652	1.387
– 14	1.811	1.515
– 13	1.984	1.653
– 12	2.172	1.803
– 11	2.376	1.964
– 10	2.597	2.139
– 9	2.837	2.328
– 8	3.097	2.532
– 7	3.379	2.752
– 6	3.685	2.990
– 5	4.015	3.246
– 4	4.372	3.521
– 3	4.757	3.817
– 2	5.173	4.136
– 1	5.623	4.479
0	6.108	4.847
1	6.566	5.192
2	7.055	5.559
3	7.575	5.947
4	8.129	6.360
5	8.719	6.797
6	9.347	7.260
7	10.01	7.750
8	10.72	8.270
9	11.47	8.819
10	12.27	9.399
11	13.12	10.01
12	14.02	10.66
13	14.97	11.35
14	15.98	12.07
15	17.04	12.83
16	18.17	13.63
17	19.37	14.48
18	20.63	15.37
19	21.96	16.31
20	23.37	17.30
21	24.86	18.34
22	26.43	19.43
23	28.09	20.58
24	29.83	21.78
25	31.67	23.05
26	33.61	24.38
27	35.65	25.78
28	37.80	27.24
29	40.06	28.78
30	42.43	30.38
31	44.93	32.07
32	47.55	33.83
33	50.31	35.68
34	53.20	37.61
35	56.24	39.63
36	59.42	41.75
37	62.76	43.96
38	66.26	46.26
39	69.93	48.67
40	73.78	51.19
41	77.80	53.82
42	82.02	56.56
43	86.42	59.41
44	91.03	62.39
45	95.86	65.50
46	100.9	68.73
47	106.2	72.10
48	111.7	75.61
49	117.4	79.26
50	123.4	83.06
51	129.7	87.01
52	136.2	91.12
53	143.0	95.39
54	150.1	99.83
55	157.5	104.4
56	165.2	109.2
57	173.2	114.2
58	181.5	119.4
59	190.2	124.7
60	199.2	130.2
61	208.6	135.9
62	218.4	141.9
63	228.5	148.1
64	293.1	154.5
65	250.1	161.2
66	261.5	168.1
67	273.3	175.2
68	285.6	182.6
69	298.4	190.2
70	311.6	198.1
71	325.3	206.3
72	339.6	214.7
73	354.3	223.5
74	369.6	232.5
75	385.5	241.8
76	401.9	251.5
77	418.9	261.4
78	436.5	271.7
79	454.7	282.3
80	473.6	293.3
81	493.1	304.6
82	513.3	316.3
83	534.2	328.3
84	555.7	340.7
85	578.0	353.5
86	601.0	366.6
87	624.9	380.2
88	649.5	394.2
89	674.9	408.6
90	701.1	423.5
91	728.2	438.8
92	756.1	454.5
93	784.9	470.7
94	814.6	487.4
95	845.3	504.5
96	876.9	522.1
97	909.4	540.3
98	943.0	558.9
99	977.6	578.1
100	1013.2	597.8
101	1050	618.0
102	1088	638.8
103	1127	660.2
104	1167	682.2
105	1208	704.7
106	1250	727.8
107	1294	751.6
108	1339	776.0
109	1385	801.0
110	1433	826.7
111	1481	853.0
112	1532	880.0
113	1583	907.7
114	1636	936.1
115	1691	965.2
116	1746	995.0
117	1804	1026
118	1863	1057
119	1923	1089
120	1985	1122
121	2049	1156
122	2114	1190
123	2182	1225
124	2250	1262
125	2321	1299
126	2393	1337
127	2467	1375
128	2543	1415
129	2621	1456
130	2701	1497
131	2783	1540
132	2867	1583
133	2953	1627
134	3041	1673
135	3131	1719
136	3223	1767
137	3317	1815
138	3414	1865
139	3512	1915
140	3614	1967

1 Sources: Smithsonian Meteorological Tables 6th. ed. (1971) and VDI vapor tables 6th ed (1963).

Table XIII: Saturation pressure p_s and vapor density ϱ_D of water in a temperature range from –100°C to +140°C[1]

Group A[3]		Group B[3]		Group C[3]	
Methane	c	Ethylene	c	Hydrogen	c
Ethane	c	Buta-1,3-diene	c	Acetylene (ethyne)	c
Propane	c	Acrylonitrile	c	Carbon bisulfide	c
Butane	c	hydrogen cyanide	a		
Pentane	c	Dethyl ether (s)	c		
Hexane	c	Ethylene oxide (oxiran)	c		
Heptane	c	1.4 Dioxan	a		
Octane	a	Tetrahydrofuran	a		
Cyclohexane	c	Tetrafluoroethylene	a		
Propylene (propene)	a				
Styrene (s)	b				
Benzene (s)	c				
Toluene (s)	–				
Xylene	a				
Naphthalene	–				
Methanol (s)	c				
Ethanol (s)	c				
Propyl alcohol (propanol)	c				
Butyl alcohol (butanol)	a				
Phenol	–				
Acetaldehyde (ethanal)	a				
Acetone (s) (propanone)	c				
Methyl ethyl ketone (s) (propan-2-one)	c				
Ethyl acetate (s)	a				
Butyl acetate (s)	c				
Amyl acetate (s)	–				
Ethyl methacrylate	–				
Acetic acid (ethanoic acid)	b				
Methyl chloride (s)	a				
Methylene chloride (s) (dichlormethane)	–				
Ammonia	a				
acetonitrile	a				
Aniline	–				
Pyridine	–				

Legend	Group A	Group B	Group C
MESG[1]	> 0.9 mm	0.5 ... 0.9 mm	< 0.5 mm
MIC[2] ratio	> 0.8 mm	0.45 ... 0.8 mm	< 0.45 mm

[1] **M**inimum **E**lectrical **S**park **G**ap
[2] **M**inimum **I**gnition **C**urrent
The ratio is based on the MIC value for laboratory methane
[3] Group allocation:
a – according to MESG value
b – according to MIC ratio
c – according to both MESG value and MIC ratio
(s) – solvent

Table XIV: Hazard classification of fluids according to their MESG1 and/or MIC2 values.
(Extract from European Standard EN 50.014)

Tables, Formulas, Diagrams

Medium x = resistant – = conditionally resistant o = not resistant	Nitrile-butadiene rubber (NBR) Perbunan	Chloroprene rubber (CR) Neoprene	Silicone rubber	Fluoro rubber (FPM, FKM) Viton	Teflon (PTFE)	EPDM
Acetaldehyde	o	o	o	o	x	x
Acetic acid (crystalline), pure	–	o	–	o	x	–
Acetic acid, industrial		x			x	
Acetic acid vapors	x	x		–	x	
Acetic acid, 20 %		x			x	x
Acetic acid, 50 %	o	x			x	x
Acetic acid, 80 %	o	o			x	
Acetic anhydride	–	x			x	x
Aceto-acetic ester				o	x	–
Acetone	o	–		o	x	x
Acetophenone	o				x	x
Acetylene	x	–			x	x
Acrylnitrile			–	o	x	
Air, clean	x	x	x	x	x	x
Air, oily	x	x	x	x	x	o
Ammonia liquid	x	x	–	–	x	x
Ammonia gas	x	x		o	x	
Amyl acetate	o	o		o	x	x
Amyl alcohol	–	–	o	o	x	x
Aniline	o	o	x		x	x
Anthracene oil	o	o	x		x	
ASTM oil No. 1	x	x	x	x	x	o
ASTM oil No. 2	x	x	x	x	x	o
ASTM oil No. 3	–	–	–	o	x	o
Benzaldehyde 100 %					x	x
Benzene	o	o		x	x	o
Benzene bromide	o	o			x	o
Benzoic acid					x	x
Bitumen					x	
Blast furnace gas	x	x	x		x	–
Boron trifluoride	x	x			x	
Bromine	o	o		x	x	
Butadiene					x	
o						
Butane	x	–		x	x	o
Butyl acetate	o	o	o		x	–
Butyl alcohol	–	x	–	o	x	x
Butyl glykol	x	x			x	
Butyraldehyde	o	o		x	x	–
Carbolineum	o	o	x	x	x	o
Carbon bisulfide	o	o	–	x	x	o

Medium x = resistant – = conditionally resistant o = not resistant	Nitrile-butadiene rubber (NBR) Perbunan	Chloroprene rubber (CR) Neoprene	Silicone rubber	Fluoro rubber (FPM, FKM) Viton	Teflon (PTFE)	EPDM
Carbon dioxide, dry	x	x	x		x	x
Carbon dioxide, wet	x	x	x		x	
Carbon tetrachloride	o	o	o	x	x	o
Chloracetic acid	o	o		o	x	x
Chlorinated solvents		o			x	
Clorine, dry					x	x
Chlorine water	–	x		o	x	x
Chlorine, wet	o	o		x	x	x
Chlorobromomethane		o			x	–
Chlorobenzene	o	o	o		x	o
Chloroform	o	o		x	x	o
Chloromethyl	o	–		x	x	o
Citrus oils	o	o			x	
Coke furnace gas	o	o			x	o
Copra oil acid	–	o	–		x	
Cottonseed oil	x	–		x	x	–
Cresol				x	x	o
Crude petroleum		–		x	x	o
Cyclohexane	x			x	x	o
Cyclohexanone	o	o	o		x	o
Cyclohexylamine					x	o
Decahydronaphtalene	x				x	o
Desmodur T	o	o			x	o
Desmophene 2000	x				x	
Dibutylphthalate	o	o	x	o	x	x
Dichlorethylene					x	
Dichlorethane			x	x	x	o
Diethylamine	–	o			x	x
Diethylene glycol	x	x			x	x
Diethyl ether	o	–	o		x	o
Diethyl ether	o	o			x	–
Diethyl sebazate	o	o			x	–
Dichlorbenzene	o	o			x	o
Dichlorbutylene	x				x	o
Diesel oil	o	–		x	x	o
Di-isopropyl ketone		o			x	x
Dimethyl ether	o				x	x
Dimethylaniline	o	o			x	
Dimethyl formamide	o	o		o	x	–
Dioctylphthalate	o	o	x		x	–

Table XV: Chemical resistance of commonly used elastomer gaskets and sealing materials

Medium x = resistant – = conditionally resistant o = not resistant	Nitrile-butadiene rubber (NBR) Perbunan	Chloroprene rubber (CR) Neoprene	Silicone rubber	Fluoro rubber (FPM, FKM) Viton	Teflon (PTFE)	EPDM
Dioxan	o	o			x	–
Diphenyl	o	o	–	x	x	o
Diphenyloxyd		o			x	x
Edenol 888				x	x	
Essential oils	o	o	–		x	o
Ethyl acetate (acetic ether)	o			o	x	x
Ethane	x	–	o	x	x	o
Ethyl acetate	o	o	o	o	x	o
Ethyl acrylate				o	x	
Ethyl alcohol, denatured	–	–		–	x	
Ethyl alcohol, pure	–	–		–	x	x
Ethyl cloride	x	–	o		x	–
Ethylene bromide	o	o			x	
Ethylene chloride					x	–
Ethylene dichloride	o	o		–	x	
Ethylene glycol	x	x	o	x	x	x
Ethyl ether	o	o		o	x	–
Ethyl silicate	x	x			x	
Ethyl acrylate					x	
Fatty acids	–	–			x	
Fatty alcohol	x	x	x		x	–
Fir leaf oil	x	o			x	
Fluorbenzene	x	o			x	o
Hydrofluoric acid, cold, 5 %	x	x		x		
Hydrofluoric acid, cold, pure	–	x				
Formaldehyde	x	–	x		x	x
Formalin, 55 %	x	x			x	x
Formic acid	–	–			x	–
Formic acid methyl ester	o	–	–		x	
Freon 11	x	x		o	x	
Freon 12	x	x		–	x	–
Freon 22	o	x		o	x	x
Freon 113	x	x		x	x	
Furane	o	o			x	o
Furfurol	o	o	o	o	x	
Gas oill	x	–		x	x	o
Generator gas	x	–	x		x	
Glycerine	x	x	x	x	x	x
Glycol	x	x	x	x	x	x
Halowax oil	o	o			x	

Medium x = resistant – = conditionally resistant o = not resistant	Nitrile-butadiene rubber (NBR) Perbunan	Chloroprene rubber (CR) Neoprene	Silicone rubber	Fluoro rubber (FPM, FKM) Viton	Teflon (PTFE)	EPDM
Heating fuel oil (coal base)	o	o		x	x	
Heating fuel oil (petroleum crude base)	x	–		x	x	o
Heptane	x	–	o	x	x	o
Hexaldehyde	o	o			x	x
Hexane	x	–	o	x	x	o
Hydraulic fluids						
Hydraulic oils DIN 51524	x	–	–	x	x	o
Phosphoric ester HFD	o	o	o		x	
Polyglycol water HFC	x	–	x	x	x	x
Hydrobromic acid	o	o			x	x
Hydrobromic crystalline acid	x				x	
Hydrocyanic acid	–	–	x		x	
Hydrogen bromide					x	
Hydrogen gas 20	x	x	–		x	
Hydrogen sulfide			x	x	x	x
Isobutyl alcohol	–	x			x	x
Isopropyl acetate	o	o			x	–
Isopropyl alcohol	–	x			x	x
Isopropyl chloride	o	o			x	o
Isopropyl ether		o			x	
Kerosene	–	–		x	x	
Kerosine	x	–		x	x	o
Lighting gas	–	–		x	x	o
Maleic anhydride					x	
Mercury	x	x			x	x
Methane	x	–			x	–
Methane (pit gas)	x	x		x	x	–
Methylene chloride	o	o		o	x	o
Methyl acrylate				o	x	o
Methyl alcohol (methanol)	–	–	x	o	x	x
Methyl ethyl ketone	o	o		o	x	–
Methyl isobutyl ketone	o			o	x	–
Methyl methacrylate	o	o		o	x	o
Methyl salicylate	o	o			x	–
Naphtalene	o	o			x	o
Natural gas	–	x	–	x	x	–
Nitrobenzene	o	o		o	x	o
Nitrous oxide	x				x	x
Oleic acid	x			x	x	o
Orange oil	o	o		x	x	

Table XV: Chemical resistance of commonly used elastomer gaskets and sealing materials

Tables, Formulas, Diagrams

Medium x = resistant – = conditionally resistant o = not resistant	Nitrile-butadiene rubber (NBR) Perbunan	Chloroprene rubber (CR) Neoprene	Silicone rubber	Fluoro rubber (FPM, FKM) Viton	Teflon (PTFE)	EPDM
Oxygen	x	x		x	x	
Ozone	o	–	x	x	x	x
Palmitic acid	x	x			x	o
Palm oil acid	–	o	x		x	
Paraffin	x	x	x		x	o
Paraffin oil	x	x			x	o
Pentachlordiphenyl	o		x		x	o
Pentane	x	x	o	x	x	o
Perchloroethylene	o	o		x	x	o
Petrol	x	–	o		x	o
Petrol alcohol 3:1	–		o	x	x	
Petrol benzene 4:1	x	o	o	x	x	o
Petrol benzene 7:3	o	o	o	x	x	o
Petrol benzene 3:2	o	o	o	x	x	o
Petrol benzene 1:1	o	o	o	x	x	o
Petrol benzene 3:7	o	o	o	x	x	
Petrol benzene spirit 5:3:2	o	o	o		x	o
Phenol	o	o	x	x	x	o
Phenyl ethyl ether	o	o			x	o
Phenylic acid (phenol)	o	o	–	–	x	–
Phosphorous chloride	o	–			x	x
Phthalic anhydride	x	x	x		x	
Piperidine	o	o			x	
Polyglycol	x				x	
Propane, gas	x	x	x	x	x	
Propylene oxyde	o				x	–
Propyl alcohol		x		x	x	x
Pydraul F-9	o	o	x	x	x	
Pydraul AC	o		x	x	x	
Pydraul A 150	o		x		x	
Pydraul A 200	o		x	x	x	
Pyridine				o	x	–
Salicylic acid	x	x			x	x
Skydrol 500			x	o	x	x
Skydrol 7000			x		x	x
Stearic acid	–				x	x
Styrene	o	o		x	x	o
Sulfur	–	x	x	x	x	x
Sulfur dioxide	o	o	x		x	x
Sulfur trioxide, dry	o	–		x	x	–

Medium x = resistant – = conditionally resistant o = not resistant	Nitrile-butadiene rubber (NBR) Perbunan	Chloroprene rubber (CR) Neoprene	Silicone rubber	Fluoro rubber (FPM, FKM) Viton	Teflon (PTFE)	EPDM
Tar oil	o				x	o
Tetrachlorethylene				x	x	o
Tetrahydrofurane	o	o			x	o
Tetraline	o	o	o		x	o
Toluene	o	o	o	x	x	o
Transformer oil	x	x		x	x	o
Train oil	o			x	x	–
Triethanolamine	o	o			x	–
Tributoxyethyl phosphate	o	o			x	o
Tributyl phosphate	o	o		o	x	o
Trichloroethane	o	o		x	x	
Trichloroethylene	o	o		x	x	o
Trichloroethyl phosphate 20	–	x			x	
Trichloroethyl phosphate 80	o	x			x	
Trichloracetic acid 60				x	x	–
Tricresyl phosphate	o		x	x	x	–
Turpentine	–	–		x	x	o
Turpentine oil, pure	x			x	x	o
Vinyl acetate	o		o		x	
Vinylaceto-acetic acid 3:2	o	–	o	o	x	
Vinyl chloride, liquid			x	x	x	
Water 50	x	x	x	x	x	
Water 100	x	–	x	x	x	
Wood oil		–			x	
Xylamon	o	o	x		x	o
Xylene	o	o	o	x	x	o

Table XV: Chemical resistance of commonly used elastomer gaskets and sealing materials

Vacuum symbols

All symbols wit the exception of those marked with **) do not depend on the position.

**) These symbols may only be used in the position shown here (tip of the angle pointing down)

The symbols for vacuum pumps should always be arranged such that the side with the constriction is allocated to the higher pressure

Vacuum pumps

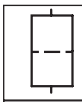
	Vacuum pump, general
	Piston vacuum pump
	Diaphragm vacuum pump
	Rotary positive displacement pump **
	Rotary plunger vacuum pump **
	Sliding vane rotary vacuum pump **
	Rotary piston vacuum pump **
	Liquid ring vacuum pump **
	Roots vacuum pump **
	Turbine vacuum pump, general
	Radial flow vacuum pump
	Axial flow vacuum pump
	Turbomolecular pump

	Ejector vacuum pump **
	Diffusion pump **
	Adsorption pump **
	Getter pump
	Sputter-ion pump
	Cryopump
	Scroll pump **
	Evaporation pump

Accessories

	Condensate trap, general
	Condensate trap with heat exchanger (e.g. cooled)
	Gas filter, general

Table XVI: Symbols used in vacuum technology (extract from DIN 28401)

	Filtering apparatus, general
	Baffle, general
	Cooled baffle
	Cold trap, general
	Cold trap with coolant reservoir
	Sorption trap
	Throttling

Vacuum chambers

	Vacuum chamber
	Vacuum bell jar

Shut-off devices

	Shut-off device, general
	Shut-off valve, straight-through valve
	Right-angle valve
	Stop cock
	Three-way stop cock

	Right-angle stop cock
	Gate valve
	Butterfly valve
	Nonreturn valve
	Safety shut-off valve

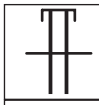
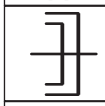
Modes of operation

	Manual operation
	Variable leak valve
	Electromagnetic operation
	Hydraulic or pneumatic operation
	Electric motor drive
	Weight-operated

Connections and piping

	Flange connection, general
	Bolted flange connection
	Small flange connection

Table XVI: Symbols used in vacuum technology (extract from DIN 28401) (continuation)

	Clamped flange connection
	Threaded tube connection
	Ball-and-socket joint
	Spigot-and-socket joint
	Taper ground joint connection
	Intersection of two lines with connection
	Intersection of two lines without connection
	Branch-off point
	Combination of ducts
	Flexible connection (e.g. bellows, flexible tubing)
	Linear-motion leadthrough, flange-mounted
	Linear-motion leadthrough, without flange
	Leadthrough for transmission of rotary and linear motion
	Rotary transmission leadthrough
	Electric current leadthrough

Measurement and gauges

	General symbol for vacuum **)
	Vacuum measurement, vacuum gauge head **)
	Vacuum gauge, operating and display unit for vacuum gauge head **)
	Vacuum gauge, recording **)
	Vacuum gauge with analog measured-value display **)
	Vacuum gauge with digital measured-value display **)
	Measurement of throughput

Table XVI: Symbols used in vacuum technology (extract from DIN 28401) (continuation)

Tables, Formulas, Diagrams

	Kelvin	Celsius	Réaumur	Fahrenheit	Rankine
Boiling point H_2O	373	100	80	212	672
Body temperature 37°C	310	37	30	99	559
Room temperature	293	20	16	68	527
Freezing point H_2O	273	0	0	32	492
$NaCl/H_2O$ 50:50	255	–18	–14	0	460
Freezing point Hg	34	–39	–31	–39	422
CO_2 (dry ice)	195	–78	–63	–109	352
Boiling point LN_2	77	–196	–157	–321	170
Absolute zero point	0	–273	–219	–460	0

	Conversion in				
	K Kelvin	°C Celsius	°R Réaumur	°F Fahrenheit	°R Rankine
K Kelvin	1	K – 273	$\frac{4}{5}$(K – 273)	$\frac{9}{5}$(K – 273) + 32	$\frac{9}{5}$K = 1,8 K
°C Celsius	°C + 273	1	$\frac{4}{5}\cdot$°C	$\frac{9}{5}\cdot$°C + 32	$\frac{9}{5}$(°C + 273)
°C Réaumur	$\frac{5}{4}\cdot$°R + 273	$\frac{5}{4}\cdot$°R	1	$\frac{9}{4}\cdot$°R + 32	$\frac{5}{9}\left[\frac{5}{4}(°R + 273)\right]$
°F Fahrenheit	$\frac{5}{9}$(°F – 32) + 273	$\frac{5}{9}$(°F – 32)	$\frac{4}{9}$(°F – 32)	1	°F + 460
°R Rankine	$\frac{5}{9}$(°R)	$\frac{5}{9}$(°R – 273)	$\frac{4}{5}\left[\frac{5}{9}(°R – 273)\right]$	°R – 460	1

Table XVII: Temperature comparison and conversion table (rounded off to whole degrees)

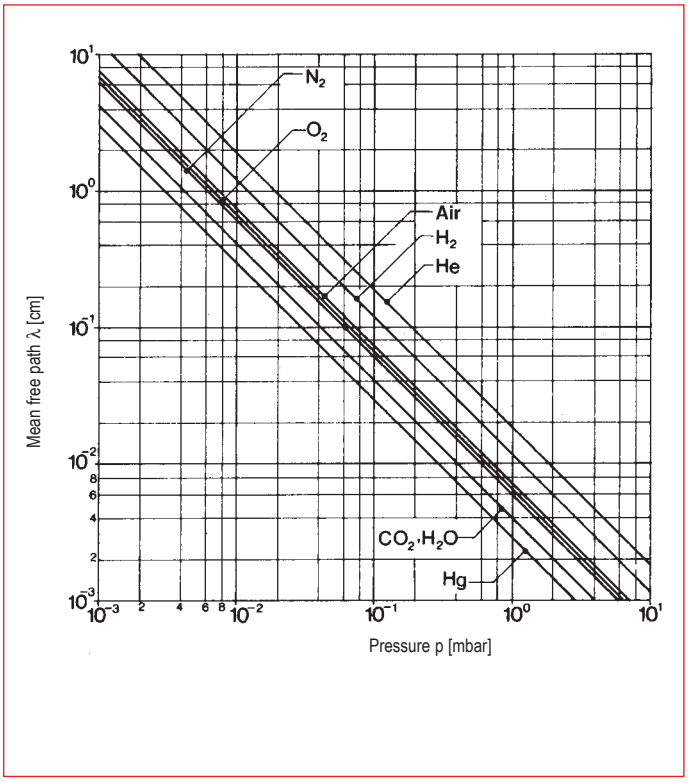
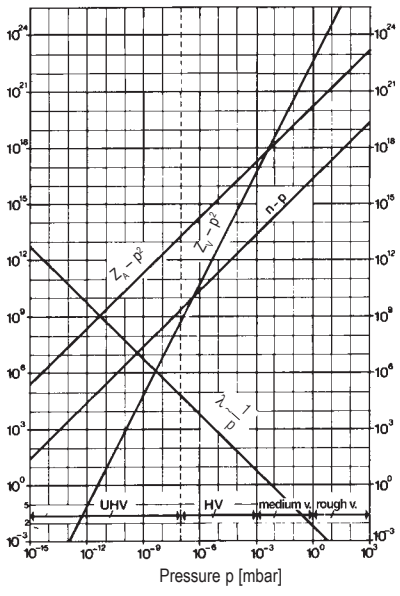


Fig. 9.1: Variation of mean free path λ (cm) with pressure for various gases



λ : mean free path in cm (λ ~ 1/p)
n : particle number density in cm^{-3} (n ~ p)
Z_A : area-related impingement rate in $cm^{-3} \cdot s^{-1}$ ($Z_A \sim p^2$)
Z_V : volume-related collision rate in $cm^{-3} \cdot s^{-1}$ ($Z_V \sim p^2$)

Fig. 9.2: Diagram of kinetics of gases for air at 20 °C

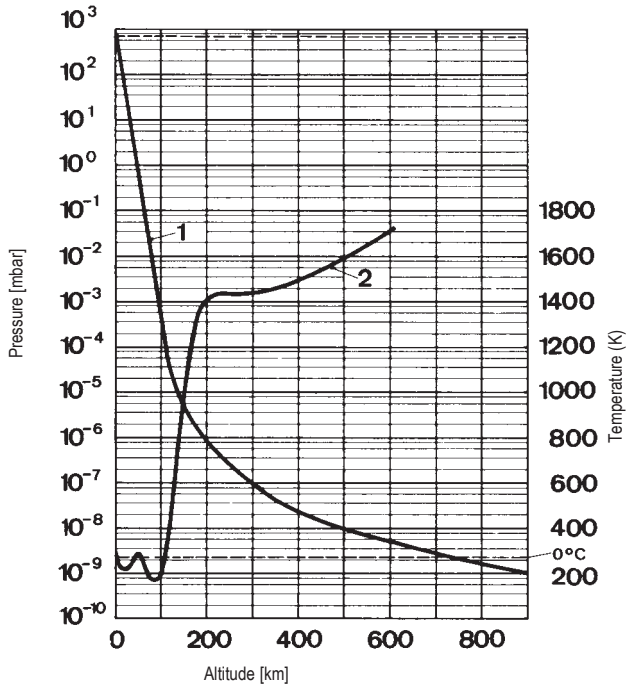


Fig. 9.3: Decrease in air pressure (1) and change in temperature (2) as a function of altitude

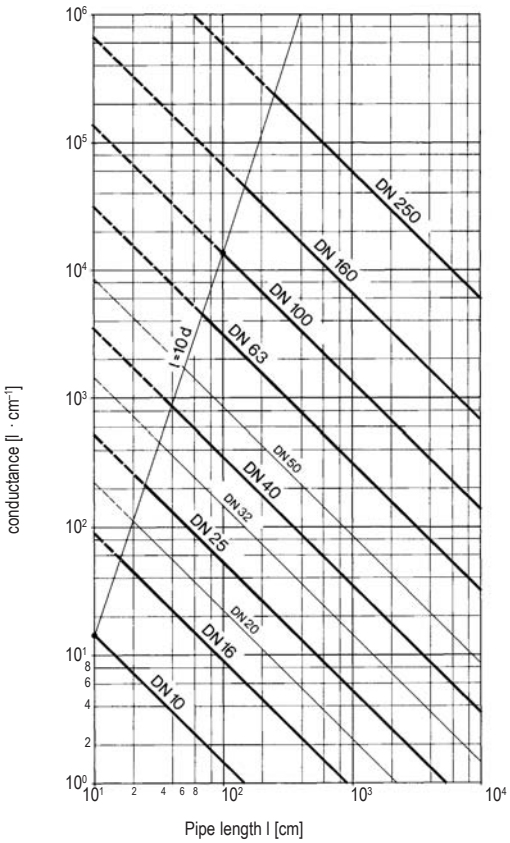


Fig. 9.5: Conductance values for piping of commonly used nominal width with circular cross-section for laminar flow (p = 1 mbar) according to equation 53a. (Thick lines refer to preferred DN) Flow medium: air (d, l in cm!)

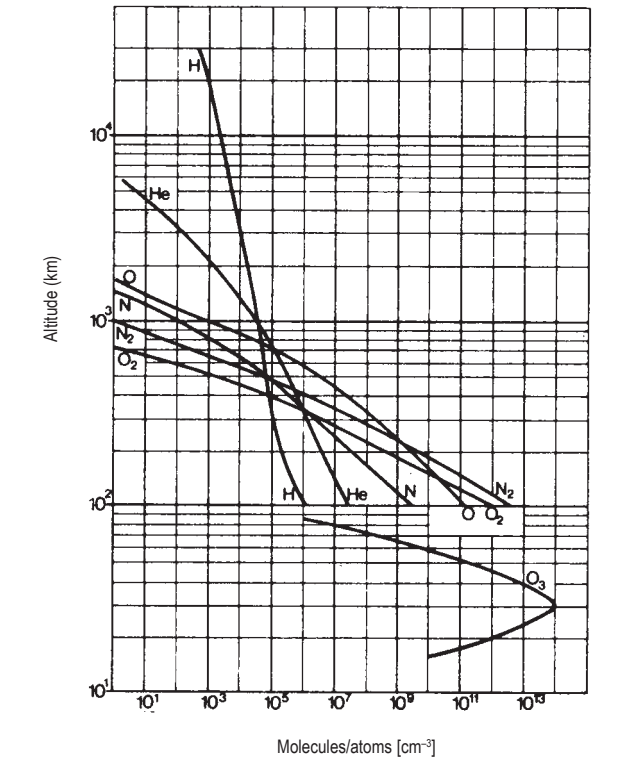


Fig. 9.4: Change in gas composition of the atmosphere as a function of altitude

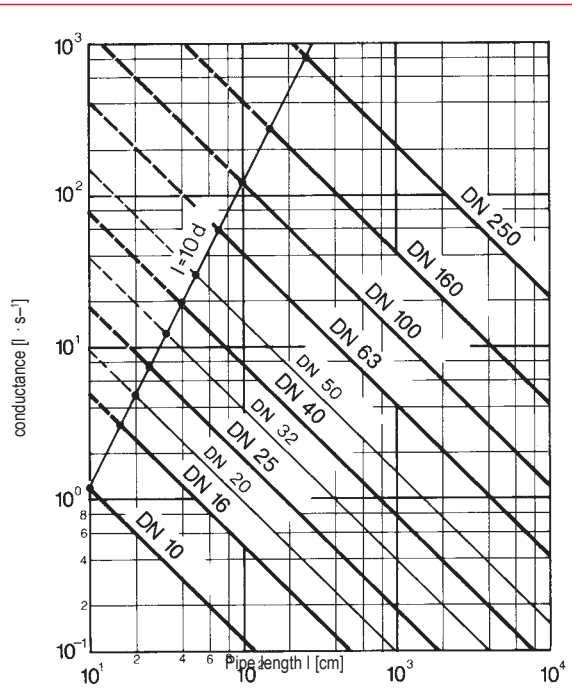
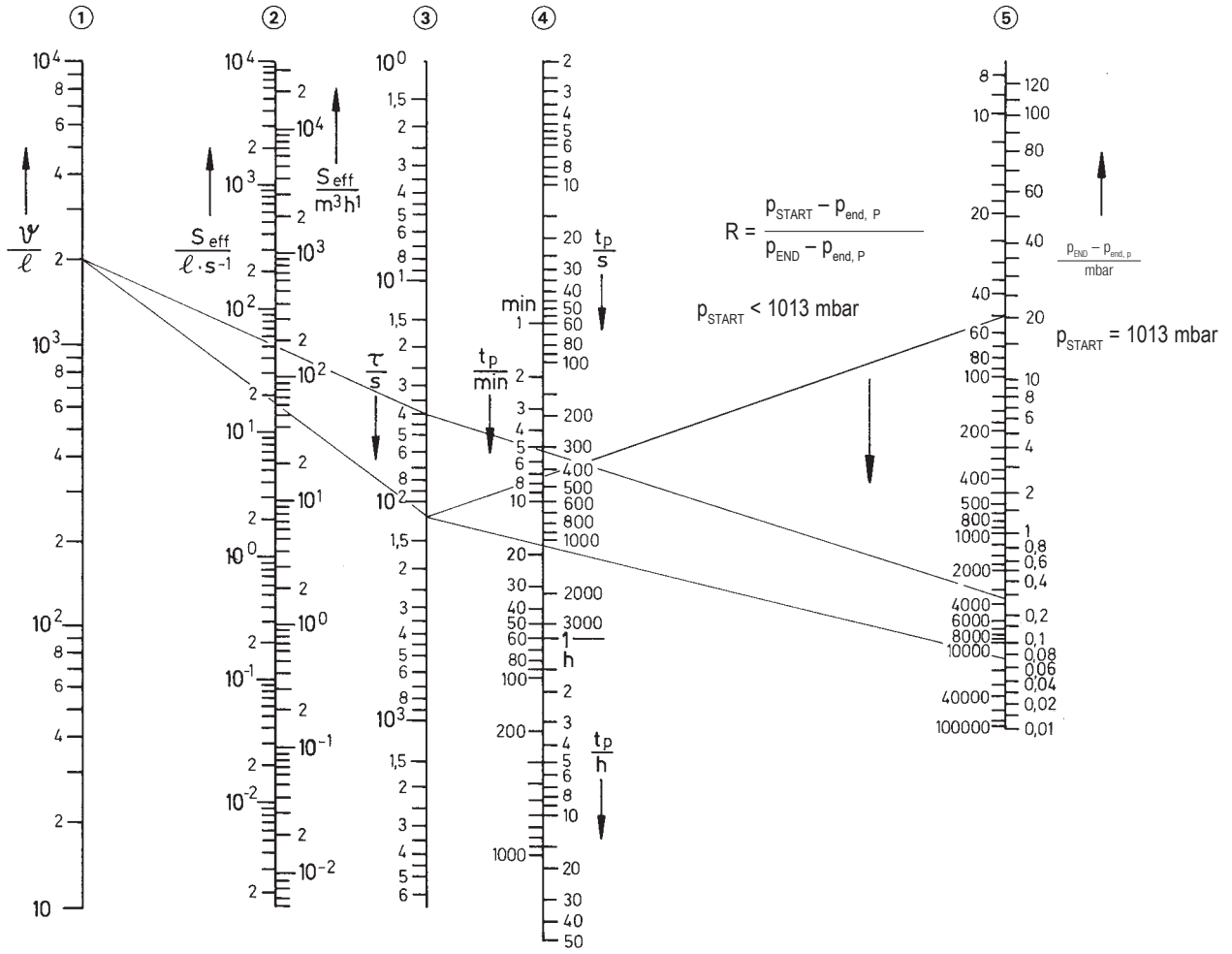


Fig. 9.6: Conductance values for piping of commonly used nominal width with circular cross-section for molecular flow according to equation 53b. (Thick lines refer to preferred DN) Flow medium: air (d, l in cm!)

Tables, Formulas, Diagrams



Column ①: Vessel volume V in liters

Column ②: Maximum effective pumping speed $S_{eff,max}$ at the vessel in (left) liters per second or (right) cubic meters per hour.

Column ③: Pump-down time t_p in (top right) seconds or (center left) minutes or (bottom right) hours.

Column ④: Right:
Pressure p_{END} in millibar at the END of the pump-down time if the atmospheric pressure p_{START} (p_n = 1013 prevailed at the START of the pump-down time. The desired pressure p_{END} is to be reduced by the ultimate pressure of the pump $p_{ult,p}$ and the differential value is to be used in the columns. If there is inflow $q_{pV,in}$, the value $p_{end} - p_{ult,p} - q_{pV,in} / S_{eff,max}$ is to be used in the columns.

Left:
Pressure reduction ratio $R = (p_{START} - p_{ult,p} - q_{pV,in} / S_{eff,max})/(p_{end} - p_{ult,p} - q_{pV,in} / S_{eff,max})$, if the pressure p_{START} prevails at the beginning of the pumping operation and the pressure is to be lowered to p_{END} by pumping down.
The pressure dependence of the pumping speed is taken into account in the nomogram and is expressed in column ⑤ $_{ult,p}$. If the pump pressure $p_{ult,p}$ is small in relation to the pressure p_{end} which is desired at the end of the pump-down operation, this corresponds to a constant pumping speed S or S_{eff} during the entire pumping process.

Example 1 with regard to nomogram 9.7:

A vessel with the volume V = 2000 l is to be pumped down from a pressure of p_{START} = 1000 mbar (atmospheric pressure) to a pressure of $p_{END} = 10^{-2}$ mbar by means of a rotary plunger pump with an effective pumping speed at the vessel of $S_{eff,max}$ = 60 m^3/h = 16.7 l $\cdot s^{-1}$. The pump-down time can be obtained from the nomogram in two steps:

1) Determination of τ: A straight line is drawn through V = 2000 l (column ① and S_{eff} = 60 m^3/h^{-1} = 16.7 l $\cdot s^{-1}$ (column ② and the value t = 120 s = 2 min is read off at the intersection of these straight lines with column ③ (note that the uncertainty of this procedure is around $\Delta\tau = \pm$ 10 s so that the relative uncertainty is about 10 %).

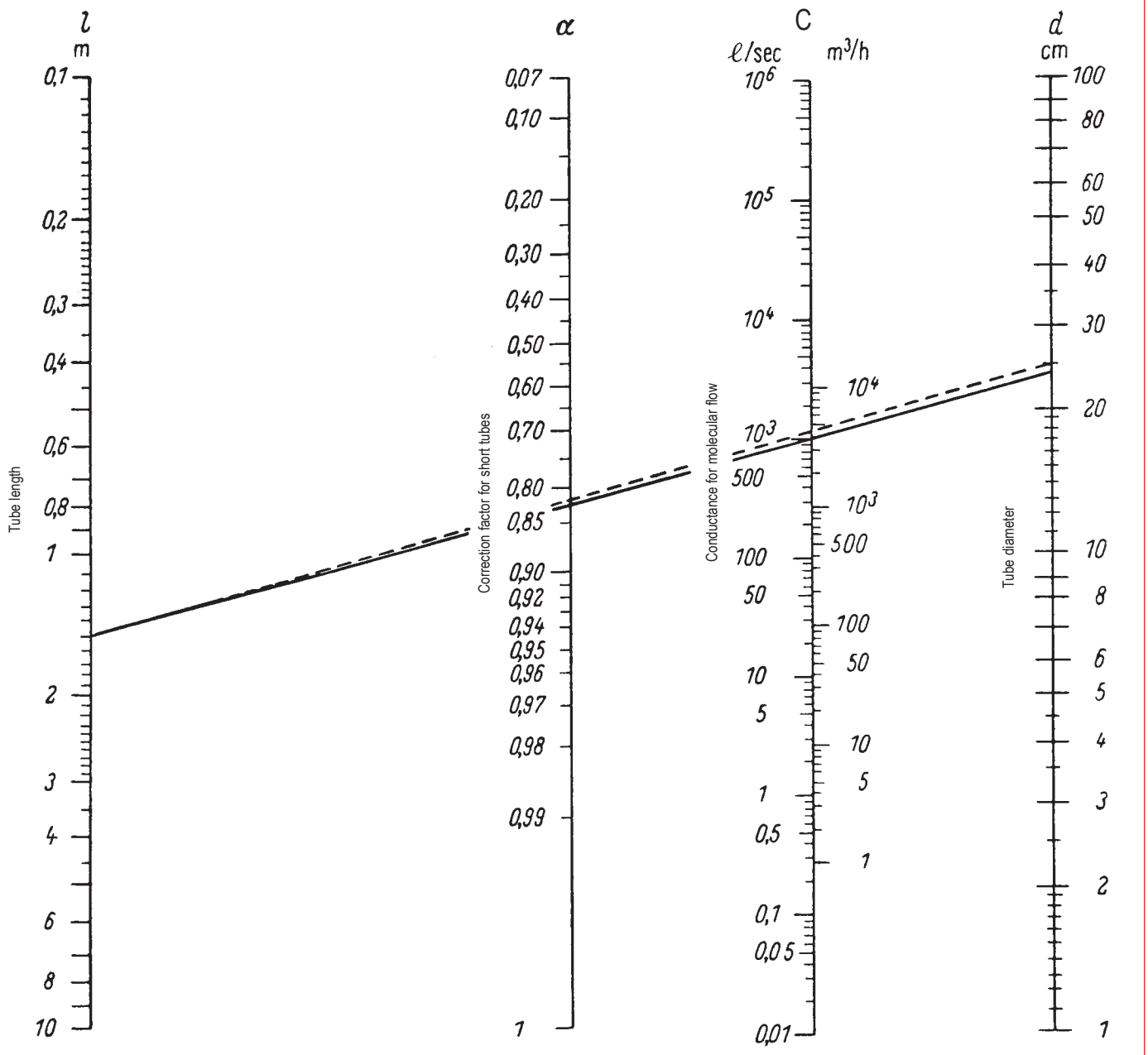
2) Determination of t_p: The ultimate pressure of the rotary pump is $p_{ult,p} = 3 \cdot 10^{-2}$ mbar, the apparatus is clean and leakage negligible (set $q_{pV,in}$ = 0); this is $p_{START} - p_{ult,p} = 10^{-1}$ mbar $- 3 \cdot 10^{-2}$ mbar $= 7 \cdot 10^{-2}$ mbar. Now a straight line is drawn through the point found under 1) τ = 120 s (column ③ and the point $p_{END} - p_{ult,p} = 7 \cdot 10^{-2}$ mbar (column ⑤ and the intersection of these straight lines with column ④ t_p = 1100 s = 18.5 min is read off. (Again the relative uncertainty of the procedure is around 10 % so that the relative uncertainty of tp is about 15 %.) Taking into account an additional safety factor of 20 %, one can assume a pump-down time of
t_p = 18.5 min · (1 + 15 % + 20 %) = 18.5 min · 1.35 = 25 min.

Example 2 with regard to nomogram 9.7:

A clean and dry vacuum system ($q_{pV,in}$ = 0) with V = 2000 l (as in example 1) is to be pumped down to a pressure of $p_{END} = 10^{-2}$ mbar. Since this pressure is smaller than the ultimate pressure of the rotary piston pump ($S_{eff,max}$ = 60 m^3/h = 16.7 l (s^{-1} = 3 $\cdot 10^{-2}$ mbar), a Roots pump must be used in connection with a rotary piston pump. The former has a starting pressure of p_1 = 20 mbar, a pumping speed of $S_{eff,max}$ = 200 m^3/h – 55 l $\cdot s^{-1}$ as well as $p_{ult,p} - 4 \cdot 10^{-3}$ mbar. From p_{start} = 1000 mbar to p = 20 mbar one works with the rotary piston pump and then connects the Roots pump from p_1 = 20 mbar to $p_{END} = 10^{-2}$ mbar, where the rotary piston pump acts as a backing pump. For the first pumping step one obtains the time constant τ = 120 s = 2 min from the nomogram as in example 1 (straight line through V = 2000 l, S_{eff} = 16.7 l $\cdot s^{-1}$). If this point in column ③ is connected with the point $p_1 - p_{ult,p} = 20$ mbar $- 3 \cdot 10^{-2}$ mbar = 20 mbar ($p_{ult,p}$ is ignored here, i.e. the rotary piston pump has a constant pumping speed over the entire range from 1000 mbar to 20 mbar) in column ⑤, one obtains $t_{p,1}$ = 7.7 min. The Roots pump must reduce the pressure from p_1 = 20 mbar to $p_{END} = 10^{-2}$ mbar, i.e. the pressure reduction ratio R = (20 mbar – 4 $\cdot 10^{-3}$ mbar) / (10^{-2} mbar-4 $\cdot 10^{-3}$) = 20/6 $\cdot 10^{-3}$ mbar = 3300.

The time constant is obtained (straight line V = 2000 l in column ①, S_{eff} = 55 l $\cdot s^{-1}$ in column ②) at = 37 s (in column ③). If this point in column ③ is connected to R = 3300 in column ⑤, then one obtains in column ④ $t_{p,2}$ = 290 s = 4.8 min. If one takes into account t_u = 1 minfor the changeover time, this results in a pump-down time of $t_p = t_{p1} + t_u + t_{p2}$ = 7.7 min + 1 min + 4.8 min = 13.5 min.

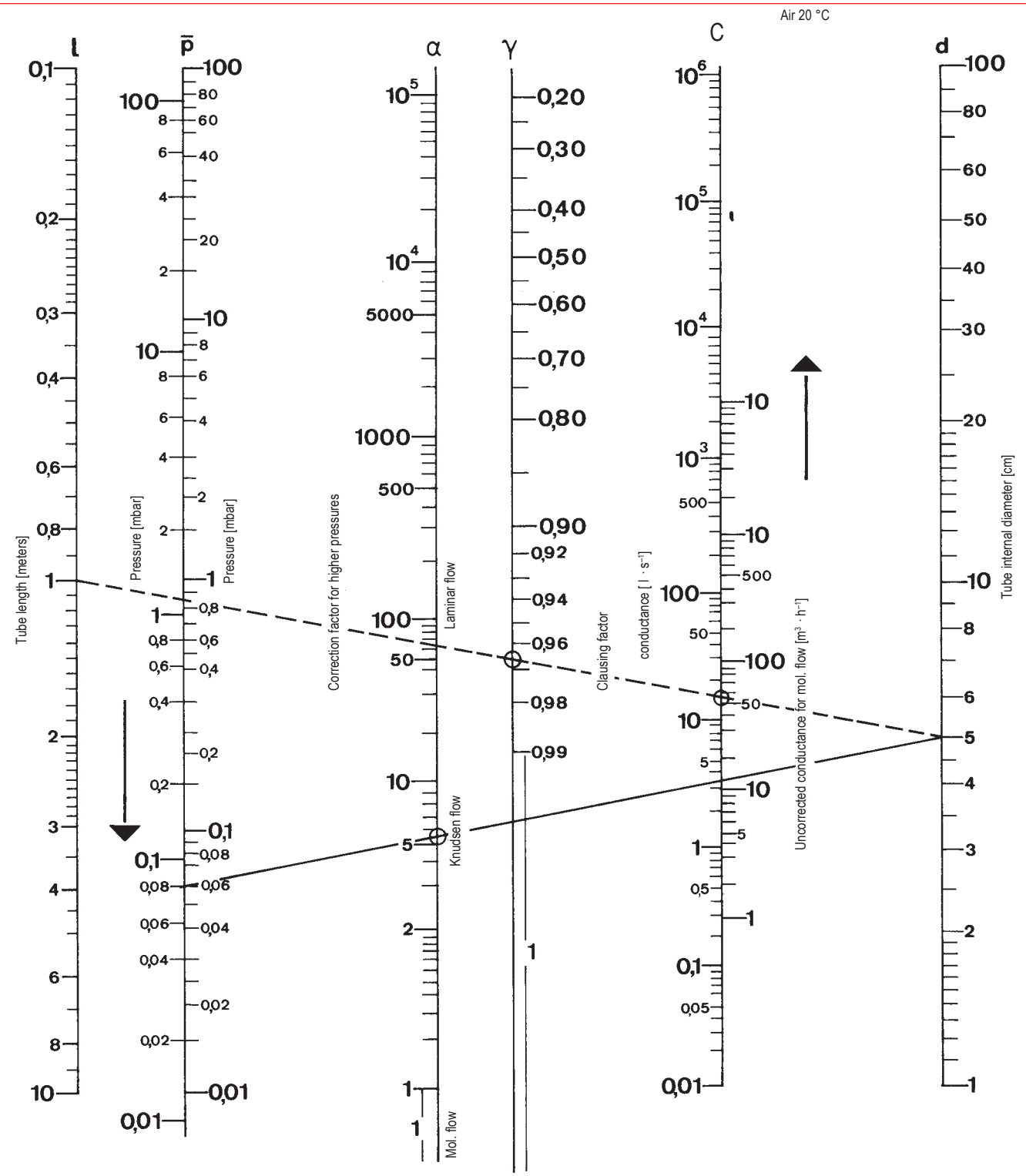
Fig. 9.7: Nomogram for determination of pump-down time tp of a vessel in the rough vacuum pressure range



Example: What diameter d must a 1.5-m-long pipe have so that it has a conductance of about C = 1000 l / sec in the region of molecular flow? The points l = 1.5 m and C = 1000 l/sec are joined by a straight line which is extended to intersect the scale for the diameter d. The value d = 24 cm is obtained. The input conductance of the tube, which depends on the ratio d / l and must not be neglected in the case of short tubes, is taken into account by means of a correction factor α. For d / l < 0.1, α can be set equal to 1. In our example d/l = 0.16 and α = 0.83 (intersection point of the straight line with the a scale). Hence, the effective conductance of the pipeline is reduced to
$C \cdot \alpha = 1000 \cdot 0.83 = 830$ l/sec. If d is increased to 25 cm, one obtains a conductance of $1200 \cdot 0.82 = 985$ l / sec (dashed straight line).

Fig. 9.8: Nomogram for determination of the conductance of tubes with a circular cross-section for air at 20 °C in the region of molecular flow (according to J. DELAFOSSE and G. MONGODIN: Les calculs de la Technique du Vide, special issue “Le Vide”, 1961).

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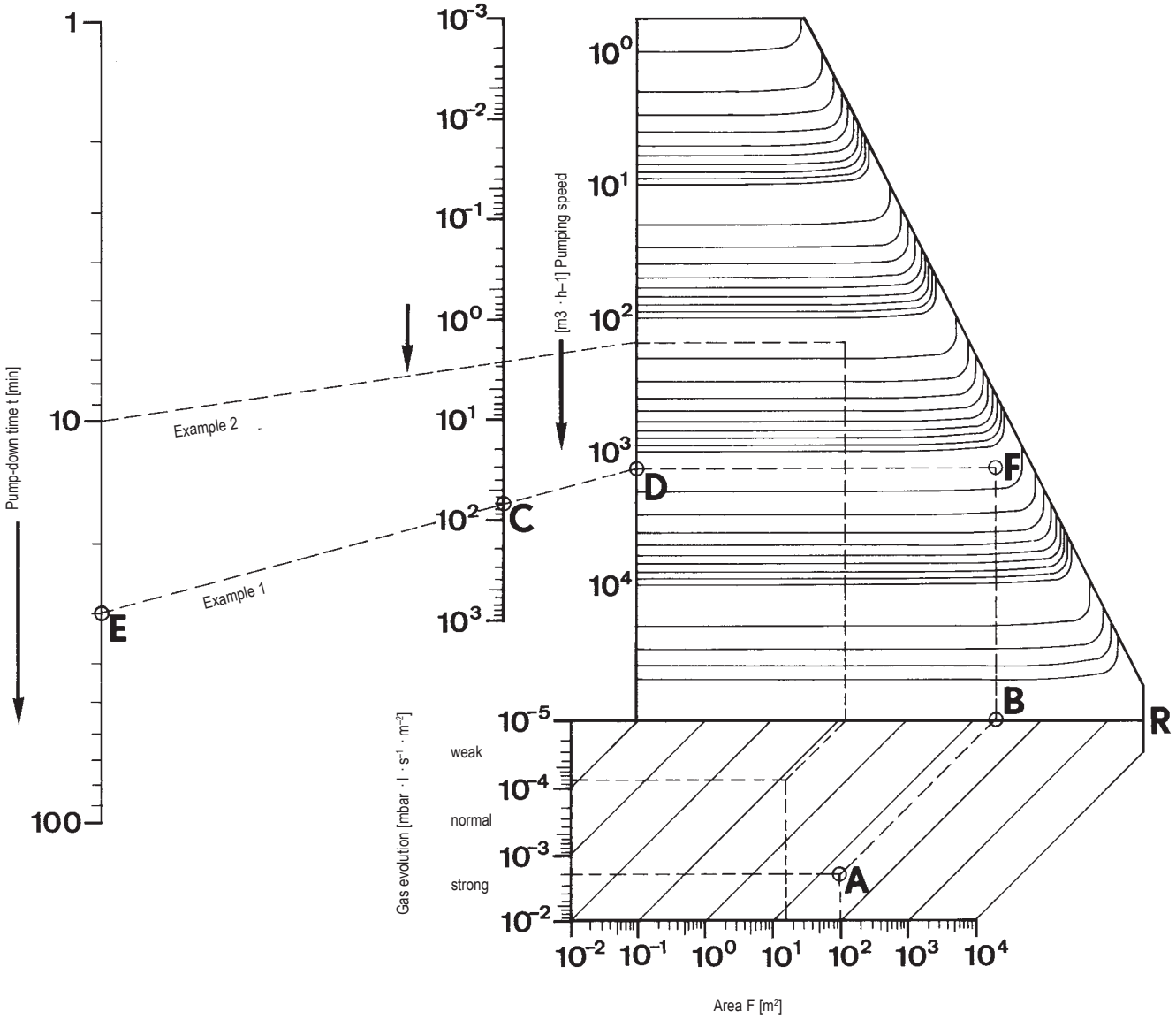


Procedure: For a given length (l) and internal diameter (d), the conductance C_m, which is independent of pressure, must be determined in the molecular flow region. To find the conductance C* in the laminar flow or Knudsen flow region with a given mean pressure of p in the tube, the conductance value previously calculated for Cm has to be multiplied by the correction factor a determined in the nomogram: $C^* = C_m \cdot \alpha$.

Example: A tube with a length of 1 m and an internal diameter of 5 cm has an (uncorrected) conductance C of around 17 l/s in the molecular flow region, as determined using the appropriate connecting lines between the "l" scale and the "d" scale. The conductance C found in this manner must be multiplied by the clausing factor $\gamma = 0.963$ (intersection of connecting line with the γ scale) to obtain the true conductance C_m in the molecular flow region:
$C_m \cdot \gamma = 17 \cdot 0.963 = 16.37$ l/s.

In a tube with a length of 1 m and an internal diameter of 5 cm a molecular flow prevails if the mean pressure p in the tube is $< 2.7 \cdot 10^{-3}$ mbar.
To determine the conductance C* at higher pressures than $2.7 \cdot 10^{-3}$ mbar, at $8 \cdot 10^{-2}$ mbar (= $6 \cdot 10^{-2}$ torr), for example, the corresponding point on the p scale is connected with the point d = 5 cm on the "d" scale. This connecting line intersects the "α" scale at the point $\alpha = 5.5$. The conductance C* at $p = 8 \cdot 10^{-2}$ mbar is: $C^* = C_m \cdot \alpha = 16.37 \cdot 5.5 = 90$ l/s.

Fig. 9.9: Nomogram for determination of conductance of tubes (air, 20 °C) in the entire pressure range.



The nomogram indicates the relationship between the nominal pumping speed of the pump, the chamber volume, size and nature of the inner surface as well as the time required to reduce the pressure from 10 mbar to 10^{-3} mbar.

Example 1: A given chamber has a volume of 70 m^3 and an inner surface area of 100 m^2; a substantial gas evolution of $2 \cdot 10^{-3}$ mbar $\cdot$ l $\cdot$ s^{-1} $\cdot$ m^{-2} is assumed. The first question is to decide whether a pump with a nominal pumping speed of 1300 m^3/h is generally suitable in this case. The coordinates for the surface area concerned of 100 m^2 and a gas evolution of $2 \cdot 10^{-3}$ mbar $\cdot$ l $\cdot$ s^{-1} $\cdot$ m^{-2} result in an intersection point A, which is joined to point B by an upward sloping line and then connected via a vertical line to the curve that is based on the pumping speed of the pump of 1300 m^3/h (D). If the projection to the curve is within the marked curve area (F), the pumping speed of the pump is adequate for gas evolution. The relevant pump-down time (reduction of pressure from 10 mbar to 10^{-3} mbar) is then given as 30 min on the basis of the line connecting the point 1300 m^3/h on the pumping speed scale to the point 70 m^3 (C) on the volume scale: the extension results in the intersection point at 30 min (E) on the time scale.

In **example 2** one has to determine what pumping speed the pump must have if the vessel (volume = approx. 3 m^3) with a surface area of 16 m^2 and a low gas evolution of $8 \cdot 10^{-5}$ mbar $\cdot$ l $\cdot$ s^{-1} $\cdot$ m^{-2} is to be evacuated from 10 mbar to 10^{-3} mbar within a time of 10 min. The nomogram shows that in this case a pump with a nominal pumping speed of 150 m^3/h is appropriate.

Fig. 9.10: Determination of pump-down time in the medium vacuum range taking into account the outgasing from the walls

Tables, Formulas, Diagrams

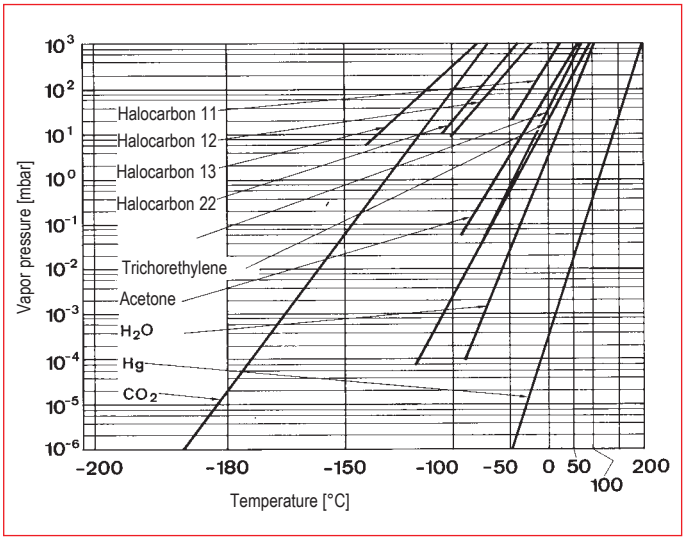


Fig. 9.11: Saturation vapor pressure of various substances

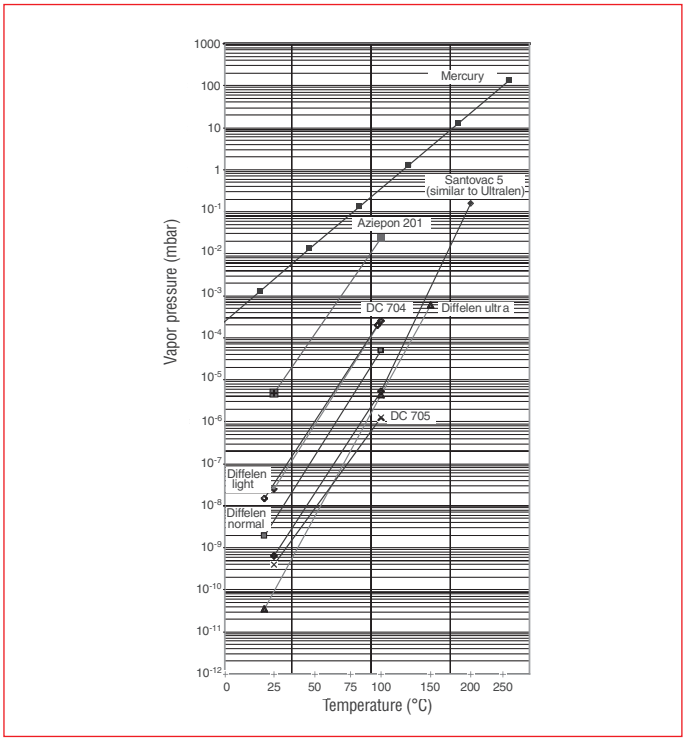


Fig. 9.12: Saturation vapor pressure of pump fluids for oil and mercury fluid entrainment pumps

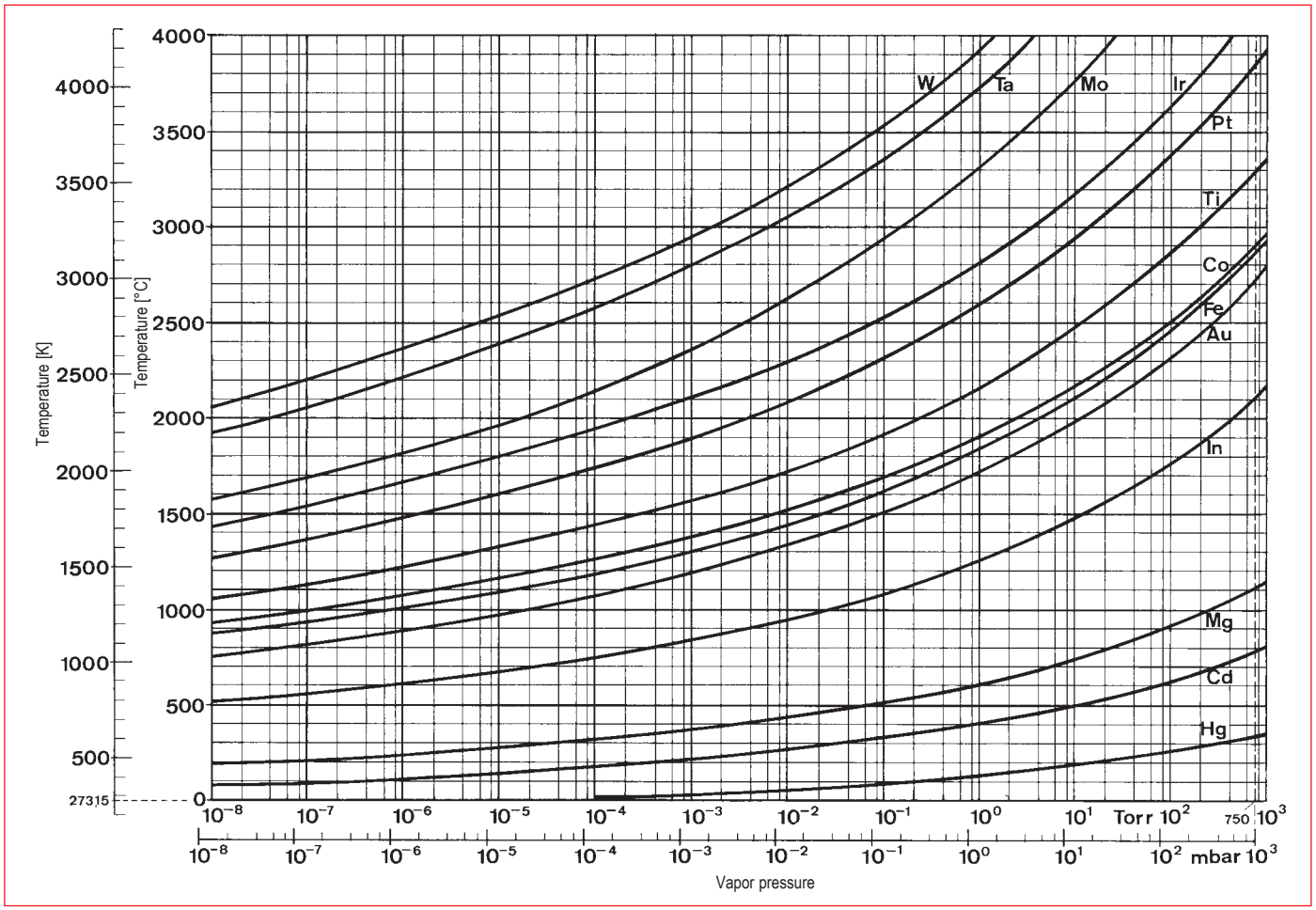
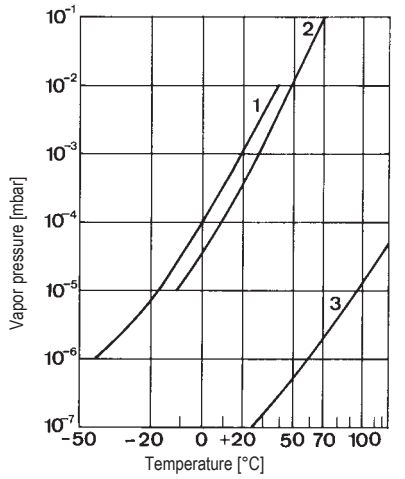


Fig. 9.13: Saturation vapor pressure of major metals used in vacuum technology



1 NBR (Perbunan) 2 Silicone rubber 3 Teflon

Fig. 9.14: Vapor pressure of nonmetallic sealing materials (the vapor pressure curve for fluoro rubber lies between the curves for silicone rubber and Teflon).

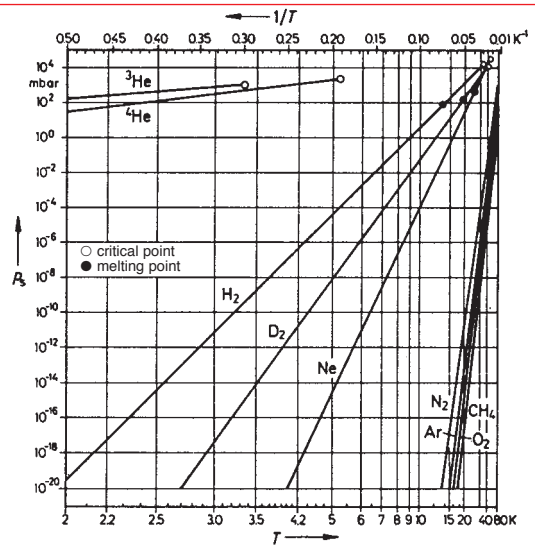


Fig. 9.15: Saturation vapor pressure p_s of various substances relevant for cryogenic technology in a temperaturerange of T = 2 – 80 K.

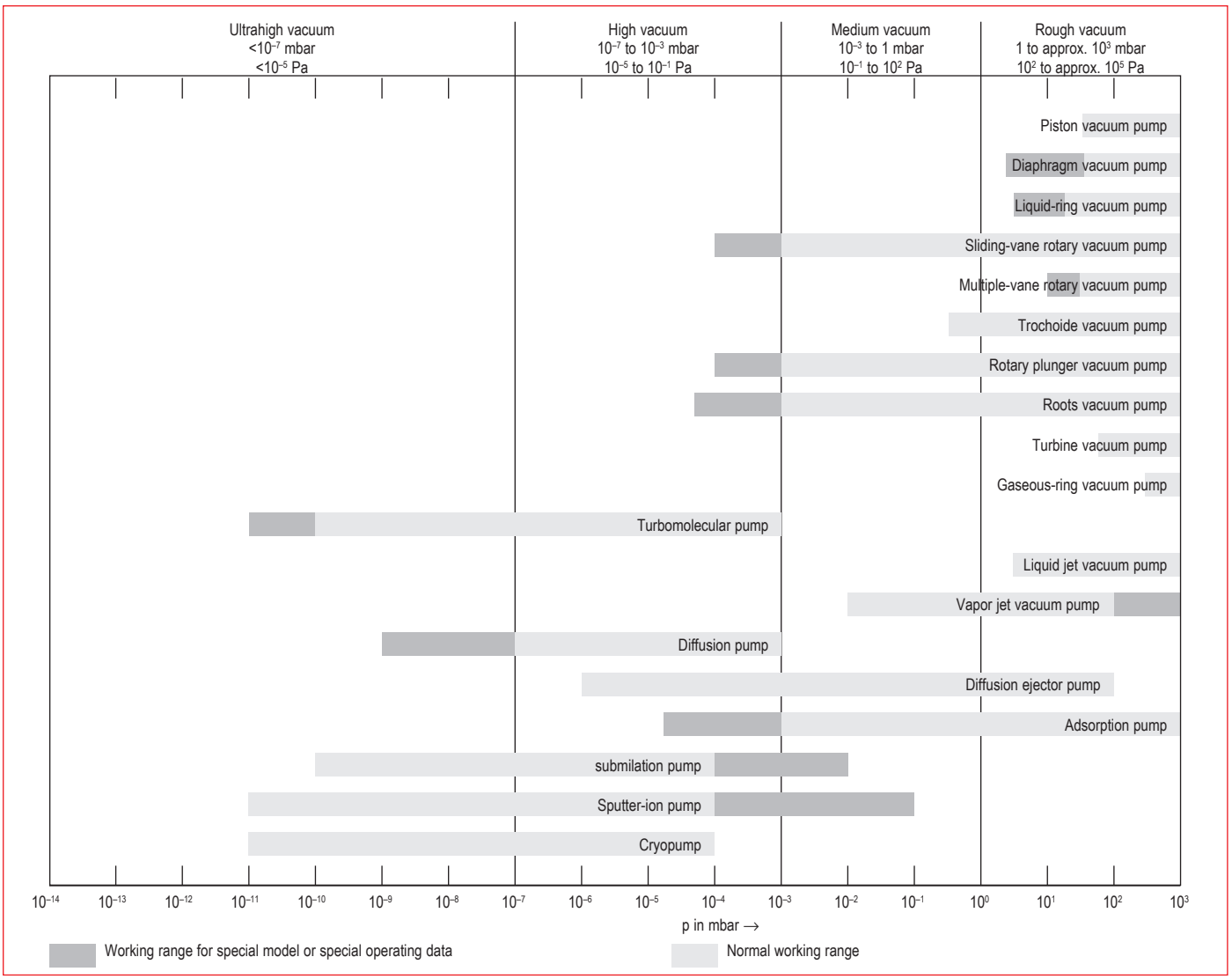


Fig. 9.16: Common working ranges of vacuum pumps

Tables, Formulas, Diagrams

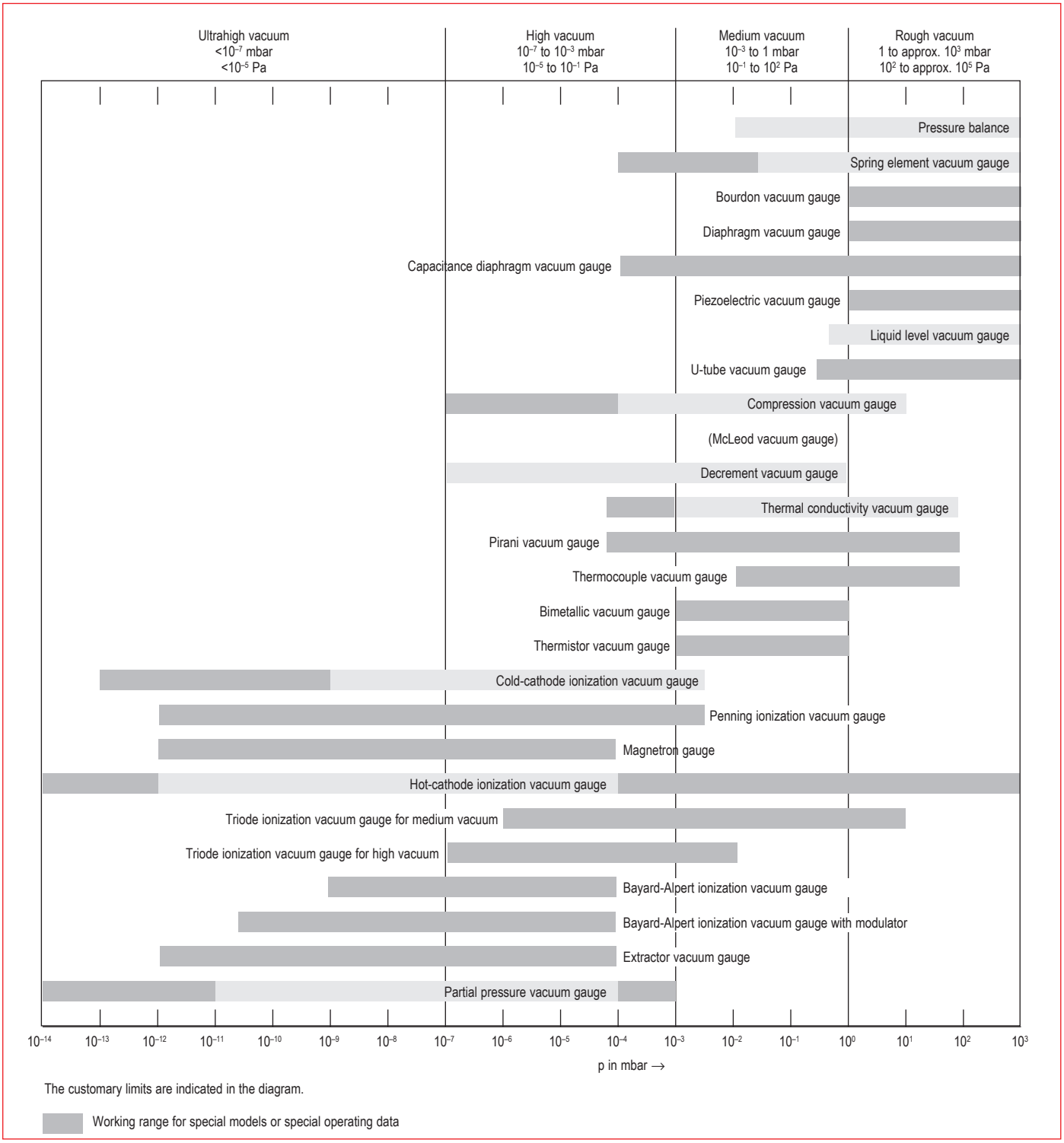


Fig. 9.16a: Measurement ranges of common vacuum gauges

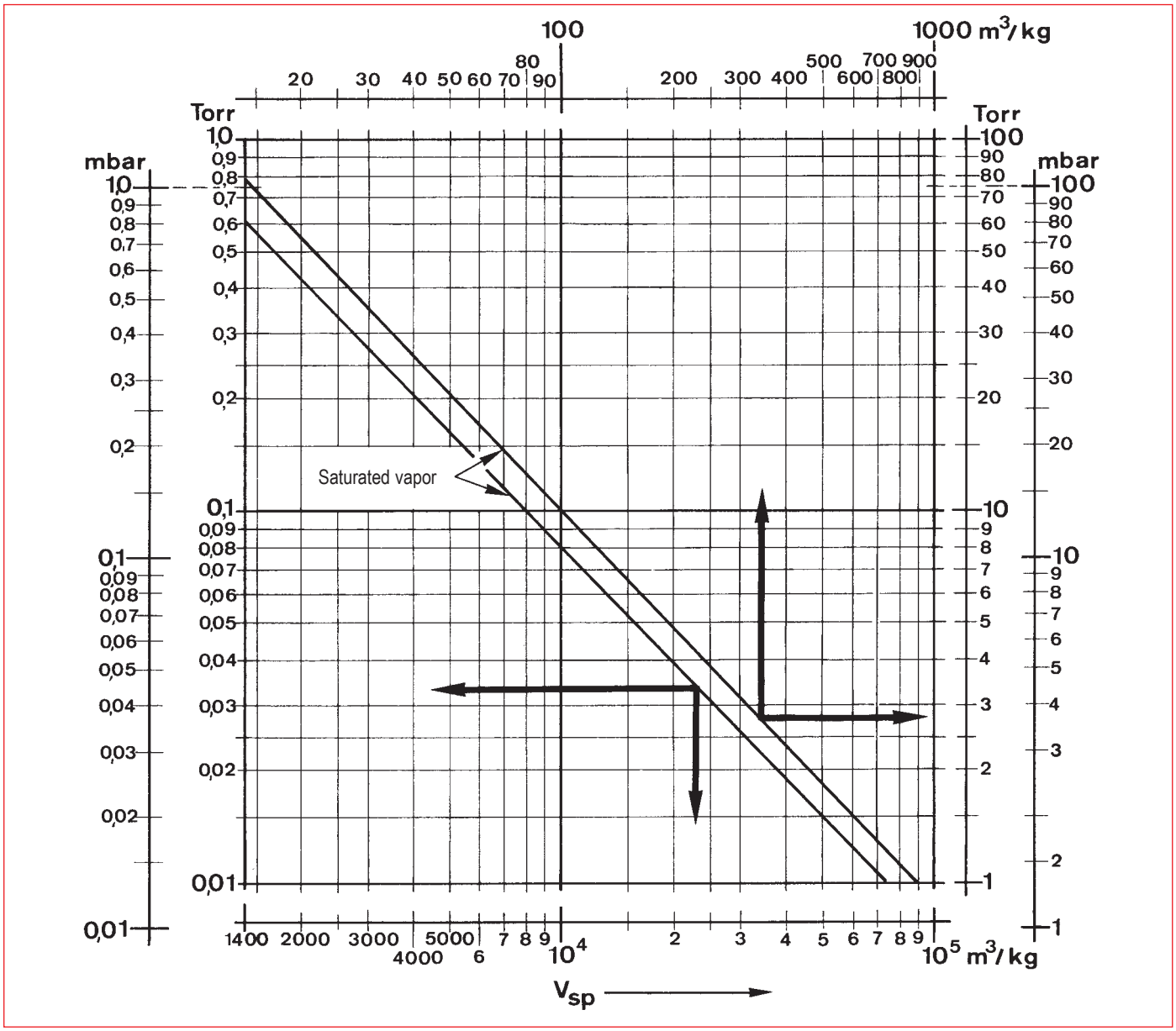


Fig. 9.17: Specific volume V_{sp} of saturated water vapor in m^3/kg within a range of 0.013 to 133 mbar.

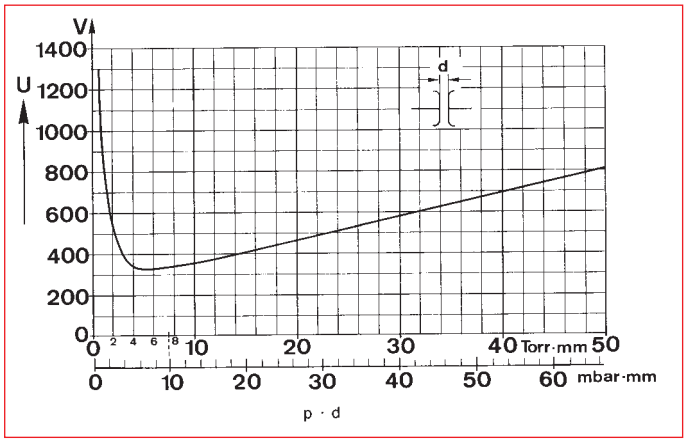


Fig. 9.18: Breakdown voltage U between parallel electrodes in a homogeneous electrical field as a function of gas pressure p distance between electrodes d (in mm) (Paschen curve), for air.

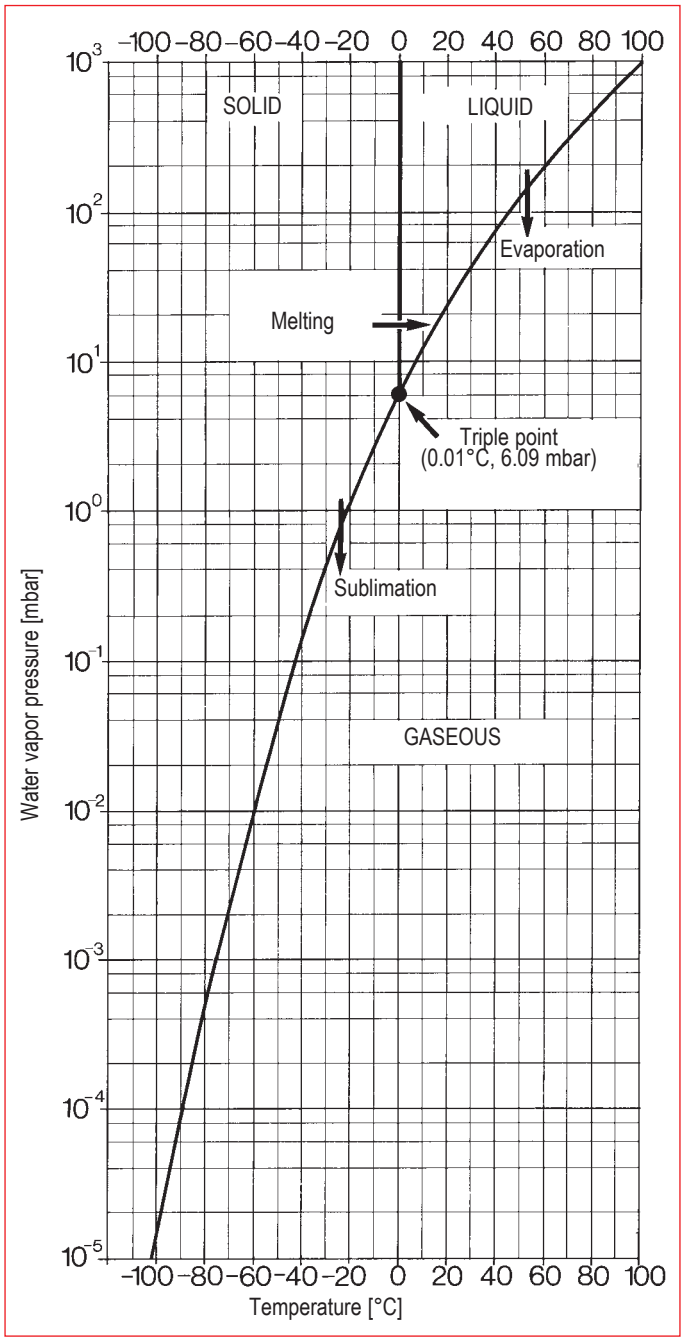


Fig. 9.19: Phase diagram of water

10. The statutory units used in vacuum technology

10.1 Introduction

Two federal German laws and the related implementing provisions stipulate which units must be used for measurements today (generally since January 1, 1978) in business and official documents and communications. The provisions resulted in a number of fundamental changes that also have to be taken into account in vacuum technology. Many of the units commonly used in the past, such as torr, gauss, standard cubic meter, atmosphere, poise, kilocalorie, kilogram-force, etc., are no longer permissible. Instead, other units are to be used, some of which are new while others were previously used in other fields. The alphabetical list in Section 10.2 contains the major variables relevant for vacuum technology along with their symbols and the units now to be used, including the SI units (see below) and legally permissible units derived from them. The list is followed by a number of remarks in Section 10.3. The purpose of the remarks is, on the one hand, to establish a connection with previous practice wherever this is necessary and, on the other hand, to provide explanations on practical use of the content of the alphabetical list.

The statutory units for measurements are based on the seven basic SI units of the Système International (SI).

Statutory units are:

a) the basic SI units (Table 10.4.1)

b) units derived from the basic SI units, in some cases with special names and unit symbols (Tables 10.4.2 and 10.4.4)

c) units used in atomic physics (Table 10.4.3)

d) decimal multiples and decimal parts of units, some with special names

Examples: 10^5 N (m^{-2} = 1 bar)
1 dm^3 = 1 l (liter)
10^3 kg = 1 t (ton)

Detailed descriptions are provided in publications by W. Haeder and E. Gärtner (DIN), by IUPAP 1987 and by S. German, P. Draht (PTB). These should always be referred to if the present summary tailored to vacuum technology leaves any questions open.

10.2 Alphabetical list[1] of variables, symbols and units frequently used in vacuum technology and its applications (see also DIN 28 402)

No.	Variable	Symbol	SI-unit	Preferred statutory units	No. of remark in Section 10.3	Notes
1	Activity (of a radioactive substance)	A	s^{-1} (B_q)	s^{-1}	3/1	
2	(General gas constant)				–	see no. 73
3	Work	W	J	J, kJ, kWh, Ws		
4	Atomic mass	m_u	kg	kg, µg		see Table V in Sect. 9
5	Avogadro constant	N_A	mol^{-1}	mol^{-1}		
6	Acceleration	a	$m \cdot s^{-2}$	$m \cdot s^{-2}$, $cm \cdot s^{-2}$		
7	Boltzmann constant	k	$J \cdot K^{-1}$	$j \cdot K^{-1}$, $mbar \cdot l \cdot K^{-1}$		see Table V in Sect. 9
8	Celsius temperature	ϑ (theta)	–	°C	3/2	
9	Vapor pressure	p_v	$N \cdot m^{-2}$, Pa	mbar, bar	3/3	Pa = Pascal
10	Time	t	s	s, min, h		see Table 10.4.4
11	Density (gas density)	ρ (ro)	$kg \cdot m^{-3}$	$kg \cdot m^{-3}$, $g \cdot cm^{-3}$	3/6	
12	Dielectric constant	ε (epsilon)	$F \cdot m^{-1}$	$F \cdot m^{-1}$, $As \cdot V^{-1} \cdot m^{-1}$		F = Farad
13	Diffusion coefficient	D	$m^2 \cdot s^{-1}$	$m^2 \cdot s^{-1}$, $cm^2 \cdot s^{-1}$		
14	Moment of momentum	L	$N \cdot s \cdot m$	$N \cdot s \cdot m$		
15	Torque	M	$N \cdot m$	$N \cdot m$, $kN \cdot m$		
16	Rotational speed, rotational frequency	n, f	s^{-1}	s^{-1}, min^{-1}		
17	Pressure in fluids	p	$N \cdot m^{-2}$, Pa	bar, mbar	3/3	Pa = Pascal

Statutory units

No.	Variable	Symbol	SI-unit	Preferred statutory units	No. of remark in Section 10.3	Notes
18	Pressure as mechanical stress	p	$N \cdot m^{-2}$, Pa	$N \cdot mm^{-2}$	3/4	
19	Diameter	d	m	cm, mm		
20	Dynamic viscosity	η (eta)	$Pa \cdot s$	$mPa \cdot s$	3/5	
21	Effective pressure	p_e	$N \cdot m^{-2}$, Pa	mbar	3/3	see also no. 126
22	Electric field strength	E	$V \cdot m^{-1}$	$V \cdot m^{-1}$		
23	Electrical capacitance	C	F	F, µF, pF		F = Farad
24	Electrical conductivity	σ (sigma)	$S \cdot m^{-1}$	$S \cdot m^{-1}$		
25	Electrical conductance	G	S	S		S = Siemens
26	Electrical voltage	U	V	V, mV, kV		
27	Electric current density	S	$A \cdot m^{-2}$	$a \cdot m^{-2}$, $A \cdot cm^{-2}$		
28	Electric current intensity	I	A	A, mA, µA		
29	Electrical resistance	R	Ω (ohm)	Ω, kΩ, MΩ		
30	Quantity of electricity (electric charge)	Q	C	C, As		C = Coulomb
31	Electron rest mass	me	kg	kg, g		see Table V in Sect. 9
32	Elementary charge	e	C	C, As		
33	Ultimate pressure	p_{ult}	$N \cdot m^{-2}$, Pa	mbar		
34	Energy	E	J	J, kJ, kWh, eV		J = Joule
35	Energy dose	D	$J \cdot k^{-1}$		3/5 a	
36	Acceleration of free fall	g	$m \cdot s^{-2}$	$m \cdot s^{-2}$		see Table V in Sect. 9
37	Area	A	m^2	m^2, cm^2		
38	Area-related impingement rate	Z_A	$m^{-2} \cdot s^{-1}$	$m^{-2} \cdot s^{-1}$; $cm^{-2} \cdot s^{-1}$		
39	Frequency	f	Hz	Hz, kHz, MHz		
40	Gas permeability	Q_{perm}	$\frac{m^3\ (NTP)}{m^2 \cdot s \cdot Pa}$	$\frac{cm^3\ (NTP)}{m^2 \cdot d \cdot bar}$	3/19	d =day (see Tab. 10.4.4 see no. 73 and no. 103)
41	Gas constant	R				
42	Velocity	v	$m \cdot s^{-1}$	$m \cdot s^{-1}$, $mm \cdot s^{-1}$, $km \cdot h^{-1}$		
43	Weight (mass)	m	kg	kg, g, mg	3/6	
44	Weight (force)	G	N	N, kN	3/7	
45	Height	h	m	m, cm, mm		
46	Lift	s	m	cm		see also no. 139
47	Ion dose	J	$C \cdot kg^{-1}$	$c \cdot kg^{-1}$, $C \cdot g^{-1}$	3/8	
48	Pulse	$\hat{p}$ (b)	$N \cdot s$	$N \cdot s$		
49	Inductance	L	H	H, mH		H = Henry
50	Isentropic exponent	κ (kappa)	–	–		$\kappa = c_p \cdot c_v^{-1}$
51	Isobaric molar heat capacity	C_{mp}	$J \cdot mol^{-1} \cdot K^{-1}$	$J \cdot mol^{-1} \cdot K^{-1}$		
52	Isobaric specific heat capacity	c_p	$J \cdot kg^{-1} \cdot K^{-1}$	$J \cdot kg^{-1} \cdot K^{-1}$		
53	Isochore molar heat capacity	C_{mv}	$J \cdot mol^{-1} \cdot K^{-1}$			
54	Isochore specific heat capacity	c_v	$J \cdot kg^{-1} \cdot K^{-1}$	$J \cdot kg^{-1} \cdot K^{-1}$		
55	Kinematic viscosity	ν (nü)	$m^2 \cdot s^{-1}$	$mm^2 \cdot s^{-1}$, $cm^2 \cdot s^{-1}$	3/9	
56	Kinetic energy	E_K	J	J		
57	Force	F	N	N, kN, mN	3/10	N = Newton
58	Length	l	m	m, cm, mm	3/11	
59	Linear expansion coefficient	α (alpha)	$\frac{m}{m \cdot K}$	$\frac{m}{m \cdot K}$; K^{-1}		
60	Leak rate	Q_L	$N \cdot m \cdot s^{-1}$	$\frac{mbar \cdot l}{s}$; $\frac{cm^3}{s}$ (NTP)	3/12	
61	Power	P	W	W, kW, mW		
62	Magnetic field strength	H	$A \cdot m^{-1}$	$A \cdot m^{-1}$	3/13	
63	Magnetic flux density	B	T	T	3/14	T = Tesla
64	Magnetic flux	Φ (phi)	Wb, $V \cdot s$	$V \cdot s$	3/15	Wb = Weber
65	Magnetic induction	B	T	T		see no. 63
66	Mass	m	kg	kg, g, mg	3/6	
67	Mass flow rate	q_m	$kg \cdot s^{-1}$	$kg \cdot s^{-1}$, $kg \cdot h^{-1}$, $g \cdot s^{-1}$		

Statutory units

No.	Variable	Symbol	SI-unit	Preferred statutory units	No. of remark in Section 10.3	Notes
68	Mass content	w_i	$kg \cdot kg^{-1}$	%, $^0/_{00}$, ppm		ppm = parts per million
69	Mass concentration	ρ_i (ro-i)	$kg \cdot m^{-3}$	$kg \cdot m^{-3}$, $g \cdot m^{-3}$, $g \cdot cm^{-3}$		
70	Moment of inertia	J	$kg \cdot m^2$	$kg \cdot m^2$		
71	Mean free path	λ	m	m, cm		
72	Molality	b_i	$mol \cdot kg^{-1}$	$mol \cdot kg^{-1}$		
73	Molar gas constant	R	$\frac{J}{mol \cdot K}$	$\frac{mbar \cdot l}{mol \cdot K}$		see Table V in Sect. 9
74	Molar mass (quantity-related mass)	M	$kg\ mol^{-1}$	$kg \cdot kmol^{-1}$, $g \cdot mol^{-1}$		
75	Molar volume	V_m	$m^3 \cdot mol^{-1}$	$m^3 \cdot mol^{-1}$, $l \cdot mol^{-1}$		
76	Molar volume, standard	V_{mn}	$m^3 \cdot mol^{-1}$	$m^3 \cdot mol^{-1}$ (NTP) $l \cdot mol^{-1}$ (NTP)		see Table V in Sect. 9
77	Molecular mass	m	kg	g		
78	Normal stress (mech.)	σ (sigma)	$N \cdot m^{-2}$	$N \cdot mm^{-2}$		
79	Standard density of a gas	ρ_n (ro-en)	$kg \cdot m^{-3}$	$kg \cdot m^{-3}$, $g \cdot cm^{-3}$		
80	Standard pressure	p_n	$N \cdot m^{-2}$, Pa	mbar		see Table V in Sect. 9
81	Standard volume	V_n	m^3	m^3 (NTP), cm^3 (NTP)	3/16	
82	Partial pressure	p_i	$N \cdot m^{-2}$, Pa	mbar	3/17	
83	Period	T	s	s, ms, µs		
84	Permeation coefficient	P	$\frac{m^3 \cdot m}{s \cdot m^2 \cdot bar}$	$\frac{cm^2}{s \cdot mbar}$	3/18	
85	Planck constant	h	$J \cdot s$	$J \cdot s$		see Table V in Sect. 9
86	pV throughput	q_{pV}	$N \cdot m \cdot s^{-1}$	$mbar \cdot l \cdot s^{-1}$	3/19	
87	pV value	pV	$N \cdot m$	$mbar \cdot l$	3/19	
88	Radius (also molecular radius)	r	m	cm, mm, µm		
89	Space charge density	ρ (ro)	$C \cdot m^{-3}$	$C \cdot m^{-3}$, $As \cdot m^{-3}$		
90	Solid angle	Ω (omega)	sr	sr		sr = steradian
91	Relative atomic mass	A_T	–	–	3/20	nondimensional variab.
92	Relative molecular mass	M_r	–	–	3/21	nondimensional variab.
93	Relative particle mass	M_r	–	–		nondimensional variab.
94	Residual vapor pressure	p_{rd}	$N \cdot m^{-2}$, Pa	mbar		
95	Residual gas pressure	p_{rg}	$N \cdot m^{-2}$, Pa	mbar		
96	Residual (total) pressure	p_r	$N \cdot m^{-2}$, Pa	mbar		
97	Reynold number nondimensional variable	Re	–	–		nondimensional variab.
98	Saturation vapor pressure	p_s	$N \cdot m^{-2}$, Pa	mbar		
99	Throughput (of a pump)	q_{pV}, Q	$N \cdot m \cdot s^{-1}$	$mbar \cdot l \cdot s^{-1}$		
100	Pumping speed	S	$m^3 \cdot s^{-1}$	$m^3 \cdot h^{-1}$, $l \cdot s^{-1}$		see no. 132
101	Stress (mech.)	ρ, σ, τ (ro, sigma, tau)	$N \cdot m^{-2}$	$N \cdot m^{-2}$, $N \cdot mm^{-2}$	3/4	see no. 18
102	Specific electron charge	$-e \cdot m_e^{-1}$	$C \cdot kg^{-1}$	$C \cdot kg^{-1}$, $As \cdot kg^{-1}$		see Table V in Sect. 9
103	Specific gas constant	R_i	$J \cdot kg^{-1} \cdot K^{-1}$	$\frac{mbar \cdot l}{kg \cdot K}$	3/22	
104	Specific ion charge	$e \cdot m^{-1}$	$C \cdot kg^{-1}$	$C \cdot kg^{-1}$, $As \cdot kg^{-1}$		
105	Specific electrical resistance	ρ (ro)	$\Omega \cdot m$	$\Omega \cdot cm$, $\Omega \cdot mm^2 \cdot m^{-1}$		
106	Specific volume	v	$m^3 \cdot kg^{-1}$	$m^3 \cdot kg^{-1}$; $cm^3 \cdot g^{-1}$		
107	Specific heat capacity	c	$J \cdot kg^{-1} \cdot K^{-1}$	$J \cdot kg^{-1} \cdot K^{-1}$, $J \cdot g^{-1} \cdot K^{-1}$	3/23	
108	Stefan-Boltzmann constant	σ (sigma)	$\frac{W}{m^2 \cdot K^4}$	$\frac{W}{m^2 \cdot K^4}$		see Table V in Sect. 9
109	Quantity of substance	ν (nü)	mol	mol, kmol		
110	Throughput of substance	q_ν	$mol \cdot s^{-1}$	$mol \cdot s^{-1}$		
111	Concentration of substance	c_i	$mol \cdot m^{-3}$	$mol \cdot m^{-3}$, $mol \cdot l^{-1}$		for substance „i“
112	Collision rate	Z	s^{-1}	s^{-1}		

Statutory units

No.	Variable	Symbol	SI-unit	Preferred statutory units	No. of remark in Section 10.3	Notes
113	Conductance	C, German: L	$m^3 \cdot s^{-1}$	$m^3 \cdot s^{-1}$, $l \cdot s^{-1}$		
114	Flow resistance	R	$s \cdot m^{-3}$	$s \cdot m^{-3}$, $s \cdot l^{-1}$		
115	Number of particles	N	–	–		nondimensional variab.
116	Particle number density (volume-related)	n	m^{-3}	cm^{-3}		
117	Particle number density (time-related)	q_N	s^{-1}	s^{-1}		see no. 120
118	Particle throughput density	j_N	$m^{-2} \cdot s^{-1}$	$m^{-2} \cdot s^{-1}$, $cm^{-2} \cdot s^{-1}$		see no. 121
119	Particle mass	m	kg	kg, g		
120	Particle flux	q_N	s^{-1}	s^{-1}		see no. 117
121	Particle flux density	j_N	$m^{-2} \cdot s^{-1}$	$m^{-2} \cdot s^{-1}$, $cm^{-2} \cdot s^{-1}$		see no. 118
122	Thermodyn. temperature	T	K	K, mK		
123	Temperature difference	ΔT, $\Delta\vartheta$	K	K, °C	3/24	
124	Temperature conductivity	a	$m^2 \cdot s^{-1}$			$a = \lambda \cdot \rho^{-1} \cdot c_p$
125	Total pressure	p_t	$N \cdot m^{-2}$, Pa	mbar	3/3	
126	Overpressure	p_e	$N \cdot m^{-2}$, Pa	mbar	3/3	
127	Ambient pressure	P_{amb}	$N \cdot m^{-2}$, Pa	mbar, bar	3/3	
128	Speed of light in vacuum	c	$m \cdot s^{-1}$	$m \cdot s^{-1}$, $km \cdot s^{-1}$		see Table V in Sect. 9
129	Evaporation heat	L_d	J	kJ		
130	Viscosity, dynamic	η (eta)	$Pa \cdot s$	$mPa \cdot s$		see no. 20
131	Volume	V	m^3	m^3, l, cm^3		
132	Volume throughput (volumetric flow)	q_v	$m^3 \cdot s^{-1}$	$m^3 \cdot h^{-1}$, $l \cdot s^{-1}$		
133	Volume concentration	σ_i (sigma-i)	$m^3 \cdot m^{-3}$	$l \cdot l^{-1}$, %, $^0/_{oo}$, ppm		ppm = parts per million
134	Volume-related collision rate	Z_v	$s^{-1} \cdot m^{-3}$	$s^{-1} \cdot m^{-3}$, $s^{-1} \cdot cm^{-3}$		
135	Quantity of heat	Q	J	J, kJ, kWh, Ws	3/25	
136	Heat capacity	C	$J \cdot K^{-1}$	$J \cdot K^{-1}$, $kJ \cdot K^{-1}$		
137	Thermal conductivity	λ (lambda)	$\frac{W}{K \cdot m}$	$\frac{W}{K \cdot m}$		
138	Heat transfer coefficient	α (alpha)	$\frac{W}{K \cdot m^2}$			
139	Path length	s	m	m, cm		
140	Wave length	λ (lambda)	m	nm	3/11	
141	Angle (plane)	α, β, γ rad (alpha, beta, gamma)	rad	rad, °, ‘, “	3/26	rad = radian
142	Angular acceleration	α (alpha)	$rad \cdot s^{-2}$	$rad \cdot s^{-2}$		
143	Angular velocity	ω (omega)	$rad \cdot s^{-1}$	$rad \cdot s^{-1}$		
144	Efficiency	η (eta)	–	–		nondimensional variab.
145	Time	t	s	s, min, h, nn, mn		see Table 10.44
146	Period of time	t, Δt	s	s, min, h		see Table 10.44

10.3 Remarks on alphabetical list in Section 10.2

3/1: Activity
The unit previously used was curie (Ci).

$1\ Ci = 3.7 \cdot 10^{10} \cdot s^{-1} = 37\ ns^{-1}$

3/2: (°C) Celsius temperature
The term degrees Celsius is a special name for the SI unit kelvin (K) [see no. 122] for indicating Celsius temperatures. The term degrees Celsius is legally approved.

3/3: Pressure
The revised version of DIN 1314 must be complied with. The specifications of this standard primarily apply to fluids (liquids, gases, vapors). In DIN 1314, bar (1 bar = 0.1 MPA = 10^5 Pa) is stated in addition to the (derived) SI unit, 1 Pa = 1 N $\cdot m^{-2}$, as a special name for one tenth of a megapascal (Mpa). This is in accordance with ISO/1000 (11/92), p. 7. Accordingly the millibar (mbar), a very useful unit for vacuum technology, is also permissible: **1 mbar = 10^2 Pa = 0.75 torr**. The unit "torr" is no longer permissible.

Special note
Exclusively absolute pressures are measured and used for calculations in vacuum technology.

In applications involving high pressures, frequently pressures are used that are based on the respective atmospheric pressure (ambient pressure) $p_{amb.}$ According to DIN 1314, the difference between a pressure p and the respective atmospheric pressure (ambient pressure) p_{amb} is designated as overpressure p_e: $p_e = p - p_{amb}$. The overpressure can have positive or negative values.

Conversions
1 kg $\cdot cm^{-2}$ = 980.665 mbar = **981 mbar**

1 at (technical atmosphere) = 980.665 mbar = **981 mbar**

1 atm (physical atmosphere) = 1013.25 mbar = 1013 mbar

1 atmosphere above atmospheric pressure (atmospheric overpressure) =

2026.50 mbar = 2 bar

$1\ torr = 1\ mm\ Hg = \frac{1\ atm}{760} = 133.322\ Pa = 1.333\ mbar$

1 meter head of water = 9806.65 Pa = 98 mbar

1 mm Hg = 133.332 Pa = 1.333 mbar = 4/3 mbar

The pressure as mechanical stress (strength) is generally given in pascal (Pa) and in N $\cdot nm^{-2}$.

Conversions:
1 Pa = 1 N $\cdot m^{-2} = 10^{-6}$ N $\cdot mm^{-2}$

1 kg $\cdot cm^{-2}$ = 98,100 Pa = 0.981 N $\cdot mm^{-2}$ = 0,1 N mm^{-2}

1 kg $\cdot mm^{-2}$ = 9,810,000 Pa = 9.81 N $\cdot mm^{-2}$ = 10 N $\cdot mm^{-2}$

3/5: Dynamic viscosity
The unit previously used was poise (P).

1 P = 0.1 Pa $\cdot$ s = 1 g $\cdot cm^{-1} \cdot s^{-1}$

3/5a: Energy dose
Rad (rd) is no longer permissible.

$1\ rd = \frac{1}{100}\ J \cdot kg^{-1}$

3/6: Weight
DIN 1305 is to be complied with in this context. Because of its previous ambivalence, the word weight should **only** be used to designate a variable of the nature of a mass as a weighing result for indicating quantities of goods.

The designations "specific weight" and "specific gravity" should no longer be used. Instead, one should say **density**.

3/7: Weight force
See DIN 1305. The previous units pond (p) and kilopond, i.e. kilogram-force, (kp) as well as other decimal multiples of p are no longer used.
1 kp = 9.81 N

3/8: Ion dose
The previously used unit was the Röntgen (R). 1 R = 2,58 $\cdot 10^{-4}$ C $\cdot kg^{-1}$

3/9: Kinematic viscosity
The previously used unit was stokes (St).

1 St = 1 $cm^2 \cdot s^{-1}$; 1 cSt = 1 $mm^2 \cdot s^{-1}$

3/10: Force
The dyne, the CGS unit for force, is no longer used.

1 dyne = 10^{-5} N

3/11: Length/wavelength
The unit Ångström (Å) (e.g. for wavelength) will no longer be used in future (see Table 4.6).

1 Å = 10^{-8} cm = 0.1 nm

3/12: Leak rate
In DIN 40.046 sheet 102 (draft of August 1973 issue), the unit mbar $\cdot dm^3 \cdot s^{-1}$ (= mbar $\cdot$ l $\cdot s^{-1}$) is used for the leak rate. Note that the leak rate corresponding to the unit 1 mbar $\cdot$ l $\cdot s^{-1}$ at 20 °C is practically the same as the leak rate 1 $cm^3 \cdot s^{-1}$ (NTP). (See also 3/17)

3/13: Magnetic field strength
The previously used unit was the oersted (Oe).

1 Oe = 79.577 A $\cdot m^{-1}$

Statutory units

3/14: Magnetic flux density
The previously used unit was the gauss (G).

1 G = 10^{-4} Vs · m^{-2} = 10^{-4} T (T = Tesla)

3/15: Magnetic flux
The previously used unit was the maxwell (M).

1 M = 10^{-8} Wb (Weber)

3/16: Standard volume
DIN 1343 must be complied with.

The designation m^3 (NTP) or m^3 (p_n, T_n) is proposed, though the expression in parentheses does not belong to the unit symbol m^3 but points out that it refers to the volume of a gas in its normal state
(T_n = 273 K, p_n = 1013 mbar).

3/17: Partial pressure
The index "i" indicates that it is the partial pressure of the "i-th" gas that is contained in a gas mixture.

3/18: Gas permeability
The **permeation coefficient** is defined as the gas flow $m^3 \cdot s^{-1}$ (volumetric flow p_V) that goes through a fixed test unit of a given area (m^2) and thickness (m) at a given pressure difference (bar).

According to DIN 53.380 and DIN 7740, Sheet 1, supplement, the gas permeability (see no. 40) is defined as "the volume of a gas, converted to 0 °C and 760 torr, which goes through 1 m^2 of the product to be tested at a certain temperature and a certain pressure differential during a day (= 24 hours)".

3/19: pV throughput/pV value
DIN 28.400, Sheet 1 is to be taken into account here. No. 86 and no. 87 have a quantitative physical significance only if the temperature is indicated in each case.

3/20: Relative atomic mass
Misleadingly called "atomic weight" in the past!

3/21: Relative molecular mass
Misleadingly called "molecular weight" in the past!

3/22: Specific gas constant
As mass-related gas constant of the substance "i". Ri = Rm (Mi^{-1}; Mi molar mass (no. 74) of the substance "i". See also DIN 1345.

3/23: Specific heat capacity
Also called specific heat:

Specific heat (capacity) at constant pressure: c_p.

Specific heat (capacity) at constant volume: c_V.

3/24: Temperature difference
Temperature differences are given in K, but can also be expressed in °C. The designation degrees (deg) is no longer permissible.

3/25: Quantity of heat
The units calorie (cal) and kilocalorie (kcal) are no longer be used.

1 kcal = 4.2 kJ

3/26: Angle
1 radian (rad) is equal to the plane angle which, as the central angle of a circle, cuts out an arc having a length of 1 m from the circle. See also DIN 1315 (8/82).

$$1° = \frac{\pi}{180} \text{ rad: } 1' = 1°/60;\ 1'' = 1'/60.$$

$$1 \text{ rad} = \frac{360°}{2\pi} \cdot 60°$$

10.4 Tables

10.4.1 Basic SI units

Basic unit	Symbol	Variable
meter	m	length
kilogramm	kg	mass
second	s	time, period; duration
ampere	A	electric current
kelvin	K	thermodyn. temperature
mole	mol	quantity of substance
candela	cd	luminous intensity

10.4.2 Derived coherent[1] SI units with special names and symbols (alphabetical)

Name of unit	Symbol	Variable	Relationship
coulomb	C	quantity of electricity or electric charge	1 C = 1 A · s
farad	F	electrical capacitance	1 F = 1 A · s · V^{-1}
henry	H	inductance	1 H = 1 V · s · A^{-1}
hertz	Hz	frequency	1 Hz = 1 · s^{-1}
joule	J	energy, work, quantity of heat	1 J = 1 N · m = Ws
lumen	lm	luminous flux	1 lm = cd · sr
lux	lx	illuminance	1 lx = 1 lm · m^{-2}
newton	N	force	1 N = 1 kgm · s^{-2}
ohm	Ω	electrical resistance	1 Ω = 1 V · A^{-1}
pascal	Pa	pressure, mechanical stress	1 Pa = 1 N · m^{-2}
radian	rad[2]	angle, plane angle	1 rad = 1 m · m^{-1}
siemens	S	electrical conductance	1 S = 1 · Ω^{-1}
steradian	sr[2]	solid angle	1 sr = 1 m^2 · m^{-2}
tesla	T	magnetic flux density or induction	1 T = 1 Wb · m^{-2}
volt	V	electrical voltage	
		or potential difference	1 V = 1 W · A^{-1}
watt	W	power, energy flux, heat flux	1 W = 1 J · s^{-1}
weber	Wb	magnetic flux	1 Wb = 1 V · s

[1] Formed with numeric factor 1; e.g. 1 C = 1 As, 1 Pa = 1 N · m^{-2}
[2] Additional SI unit

10.4.3 Atomic units

Basic unit	Symbol	Vartiable
Atomic mass unit	m_u	Mass for indication of particle mass; 1 m_u = 1/12 mass of ^{12}C also amu (atomic mass unit).
Electron volt	eV	energy

10.4.4 Derived noncoherent SI units with special names and symbols

Basic unit	Symbol	Definition
Day	d	1 d = 86.400 s
Hour	h	1 h = 3.600 s
Minute	min	1 min = 60 s
Round angle	–	2 π rad
Degree	(°)	$\frac{\pi}{180}$ rad
Minute	(')	$\frac{\pi}{10.800}$ rad (= $\frac{1}{60}$grad)
Second	(")	$\frac{\pi}{648.000}$ rad (= $\frac{1}{60}$minute)

Vacuum technology standards

11. National and international standards and recommendations particularly relevant to vacuum technology

For around 20 years now, numerous standards and recommendations have been drawn up at national and international level and revised, whenever necessary, in accordance with the state of the art. These standards and recommendations must be observed whenever use is made of vacuum equipment (pumps, gauges, valves, etc.) and vacuum apparatus, systems and plants are assembled. They not only contain specifications applying specially to vacuum technology, but also go beyond this specific field and involve, for example, physical units, formulas, noise protection regulations, etc.

National standards are primarily DIN standards, particularly those relating to the area of vacuum technology in the DIN Standards Committee on Mechanical Engineering (NAM). International standards and recommendations are drawn up and issued

a) by the International Standardization Organization (ISO), in particular by ISO Committee TC 112 (vacuum technology)

b) by the European Committee of Manufacturers of Compressors, vacuum pumps and compressed air tools (PNEUROP), in particular by PNEUROP Subcommittee C5 (vacuum technology)

c) by the European Committee for Standardization (CEN), in particular by Technical Committee TC 138 (nondestructive testing) and Technical Committee TC 318.

The content of the documents drawn up by the international organizations in a) to c), with German participation (also by LEYBOLD), has been extensively incorporated into DIN standards, as reflected in designations such as DIN / ISO or DIN / EN.

The most important standards to be complied with are listed in Table 11.1 below.

Abbreviations used:
D = draft
CD = Committee Draft

11.1 National and international standards and recommendations of special relevance to vacuum technology

A) National agreements, Part 1: DIN

DIN	Title	Issue
1301	Units	
	Part 1 – Names of units, symbols	1993
	Part 2 – Parts and multiples generally used	2/78
	Part 3 – Conversions for units no longer used	6/79
1304	General symbols	
	Part 1 – General symbols	3/94
	Part 2 – Symbols for meteorology and geophysics	9/89
	Part 3 – Symbols for electrical energy supply	3/89
	Part 5 – Symbols for flow mechanics	9/89
	Part 6 – Symbols for electrical communications technology	5/92
	Part 7 – Symbols for meteorology and geophysics	
	Part 7 – Symbols for electric machines	1/91
1305	Mass; weighed value, force, weight force, weight load, definitions	1/88
1306	Density; definitions	6/84
1313	Physical variables and equations, definitions, spelling	4/78
1314	Pressure; basic definitions, units	2/77
1319	Basic definitions for measurement technology	
	Part 1 – Basic definitions	1/95
	Part 2 – Definitions for the use of gauges	1/80
	Part 3 – Definitions for measurement uncertainty and evaluation of gauges and measuring equipment	8/83
	Part 4 – Treatment of uncertainty in the use of measurements	12/85

A) National agreements, Part 1: DIN (cont.)

DIN	Title	Issue
1343	Normal state, reference state	1/90
1345	Thermodynamics; basic definitions	12/93
1952	Flow measurement with screens, nozzles, etc.	7/82
2402	Pipelines; nominal internal diameters, definitions, classification	2/76
3535	Seals for gas supply – Part 6	4/94

Vacuum technology standards

DIN	Title	Issue
8964	Circuit parts for refrigeration systems with hermetic and semi-hermetic compressors Part 1: Tests Part 2: Requirements	 3/96 9/86 (E 12/95)
16005	Overpressure gauges with elastic measuring element for general use Requirements and testing	2/87
16006	Overpressure gauges with Bourdon tube Safety-related requirements and testing	2/87
19226	– 1 Control and instrumentation technology; control and regulation technology; definitions – general principles – 4 Control and instrumentation technology; control and regulation technology; definitions for control and regulation systems – 5 Control and instrumentation technology; control and regulation technology; functional definitions	2/94 2/94 2/94
25436	Integral leak rate test of safety vessel with absolute pressure method	7/80
28090	Static seals for flange connections Part 1 - Characteristic values for seals and testing methods Part 2 - Seals made of sealing plate; special testing methods for quality assurance	 9/95 9/95
28400	Vacuum technology; designations and definitions Part 1 - Basic definitions, units, ranges, characteristic variables and basic principles Part 2 - Vacuum pumps Part 3 - Vacuum plants; characteristic variables and gauges Part 4 - Vacuum coating technology Part 5 - Vacuum drying and vacuum freeze-drying Part 6 - Analytical techniques for surface technology Part 7 - Vacuum metallurgy Part 8 - Vacuum systems, components and accessories	 5/90 10/80 6/92 3/76 3/81 10/80 7/78 10/80 (E 7/91)
28401	Vacuum technology; symbols – overview	11/76
28402	Vacuum technology: variables, symbols, units - overview	12/76
28403	Vacuum technology; quick connections, small flange connections	9/86
28404	Vacuum technology: flanges, dimensions	10/86

DIN	Title	Issue
28410	Vacuum technology; mass spectrometer partial pressure gauges, definitions, characteristic variables, operating conditions	11/86
28411	Vacuum technology; acceptance specifications for mass spectrometer leak detection devices, definitions	3/76
28416	Vacuum technology; calibration of vacuum gauges in a range from 10^{-3} to 10^{-7} mbar. General methods; pressure reduction through constant flow	3/76
28417	Vacuum technology; measuring pV mass flow according to volumetric method at constant pressure	3/76
28418	Vacuum technology; standard method for calibrating vacuum gauges through direct comparison with a reference device Part 1 – General principles Part 2 – Ionization vacuum gauges Part 3 – Thermal conductivity vacuum gauges	 5/76 9/78 8/80
28426	Vacuum technology; acceptance specifications for rotary piston vacuum pumps Part 1 – Rotary piston and vane type rotary vacuum pumps in rough and medium vacuum range Part 2 – Roots vacuum pumps in medium vacuum range	
28427	Vacuum technology; acceptance specifications for diffusion pumps and ejector vacuum pumps for pump fluid vapor pressures of less than 1 mbar	2/83
28428	Vacuum technology; acceptance specifications for turbo-molecular pumps	11/78
	28429 Vacuum technology; acceptance specifications for getter-ion pumps	8/85
28430	Vacuum technology; measuring specifications for ejector vacuum pumps and ejector compressors. Pump fluid: water vapor	11/84
28431	Acceptance specifications for liquid ring vacuum pumps	1/87
28432	Acceptance specifications for diaphragm vacuum pumps	E 5/95
53380	Testing of plastic foils, determination of gas permeability	6/69 (E 10/83)
45635	Noise measurement at machines: measurement of airborne noise, enveloping surface methods. Part 13 – Compressors, including vacuum pumps, positive displacement, turbo and steam ejectors	2/77

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DIN	Title	Issue
55350	Definitions of quality assurance and statistics Part 11 – Basic definitions of quality assurance Part 18 – Definitions regarding certification of results of quality tests/quality test certificates	8/95 7/87
66038	Torr – millibar; millibar – torr conversion tables	4/71
–	Thesaurus Vacui (definition of terms)	1969

A) European/national agreements, EN, DIN/EN, CEN

DIN/EN	Title	Issue
EN 473	Training and certification of personnel for nondestructive testing (including leak test)	7/93
837-1	Pressure gauges, Part 1: Pressure gauges with Bourdon tubes, dimensions, measurement technology, requirements and testing	2/97
837-2	Pressure gauges, Part 2: Selection and installation recommendations for pressure gauges	1/95
837-3	Pressure gauges, Part 3: Pressure gauges with plate and capsule elements, dimensions, measurement technology, requirements and testing	2/97
1330-8 E	Nondestructive testing - definitions for leak test – terminology	6/94
1779 E	Nondestructive testing – leak test. Instructions for selection of a testing method	3/95
1338-8 E	Nondestructive testing – leak test. Terminology on leak test	1994
1518 E	Nondestructive testing – determination of characteristic variables for mass spectrometer leak detectors	
1593 E	Nondestructive testing – bubble type testing method	12/94
NMP 826 Nr. 09–95	Calibration of gaseous reference leaks, CD	9/95

B) International agreements, ISO, EN/ISO

ISO	Title	Issue
1000	SI units and recommendations for the use of their multiples and of certain other units	11/92
1607 / 1	Positive displacement vacuum pumps. Measurement of performance characteristics. Part 1 - Measurement of volume rate of flow (pumping speed)	12/93
1607 / 2	Positive displacement vacuum pumps. Measurement of performance characteristics. Part 2 - Measurement of ultimate pressure	11/89
1608 / 1	Vapor vacuum pumps. Part 1: Measurement ofvolume rate of flow	12/93
1608 / 2	Vapor vacuum pumps. Part 2: Measurement of critical backing pressure	12/89
1609	Vacuum technology. Flange dimensions	3/86
DIN/ISO 2533	Standard atmosphere	12/79
2861 / 1	Quick release couplings. Dimensions Part 1 - Clamped Type	8/74
2861 / 2	Quick release couplings. Dimensions Part 2 - Screwed type	8/80
3529 / 1	Vacuum Technology Vocabulary Part 1 - General terms	12/81
3529 / 2	Vacuum Technology Vocabulary Part 2 - Vacuum pumps and related terms	12/81
3529 / 3	Vacuum Technology Vocabulary Part 3 - Vacuum gauges	12/81
3556 / 1	Measurement of performance characteristics. Part 1 - Sputter ion pumps (E)	1992
3567	Vacuum gauges. Calibration by direct comparison with a reference gauge (CD)	2/91
3568	Ionisation vacuum gauge. Calibration by direct comparison with a reference gauge (CD)	2/91
3570 / 1	Vacuum gauges – standard methods for calibration Part 1 - Pressure reduction by continuous flow in the pressure range 10^{-1} ... 10^{-5} Pa.	2/91
3669	Vacuum Technology. Bakable flanges, dimensions. Part 1: Clamped Type	2/86
EN/ISO 4080	Rubber and plastic hoses and hose lines – determination of gas permeability	4/95
5167	Measurement of fluid flow by means of orifice plates, nozzles etc.	1980
5300	Vacuum gauges of the thermal conductivity type. Calibration by direct comparison with a reference gauge (CD)	2/91
9803	Pipeline fittings-mounting, dimensions (E)	2/93
DIN/ISO 10012	Requirements placed on quality assurance for measuring equipment Part 1 – Confirmation system for measuring equipment	8/92

C) PNEUROP/C5 (6.93)

Number	Title/remark	identical to DIN	Issue
5607	Vacuum pumps; acceptance specifications Part II: (Fluid entrainment pumps)	28427	1972
5608	Vacuum pumps; acceptance specifications Part III: (Turbomolecular pumps)	28428	1973
5615	Vacuum pumps; acceptance specifications Part IV: (Getter-ion pumps)	28429	1976
6601	Measurement of performance of ejector vacuum pumps and ejector compressors	28430	5/78
6602	Vacuum pumps; acceptance specifications Part I: (Oil-sealed rotary pumps and Roots pumps)	28426	1979
6606	Vacuum flanges and connections; dimensions	28403 and 28404	1985
PN5ASR CC/5	Vacuum pumps, acceptance specifications refrigerator cooled cryopumps		7/89

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