

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 conductance 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

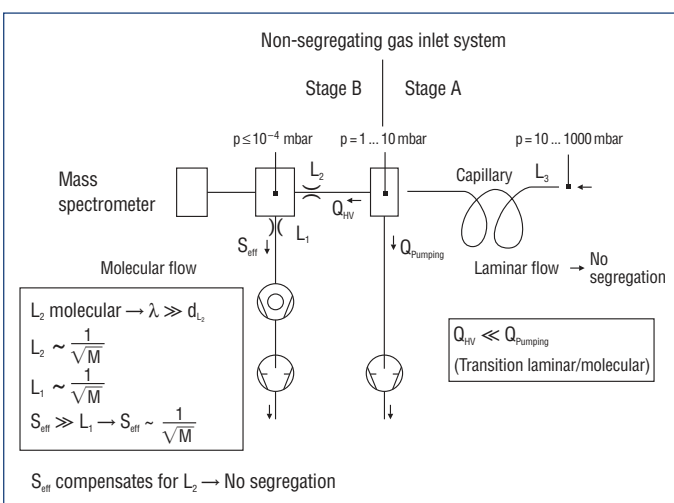


Fig. 4.7 Principle of the pressure converter (stage B only in the single-stage version and stages A and B in two-stage units)

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).

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-CO₂ 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 H₂O⁺ 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.

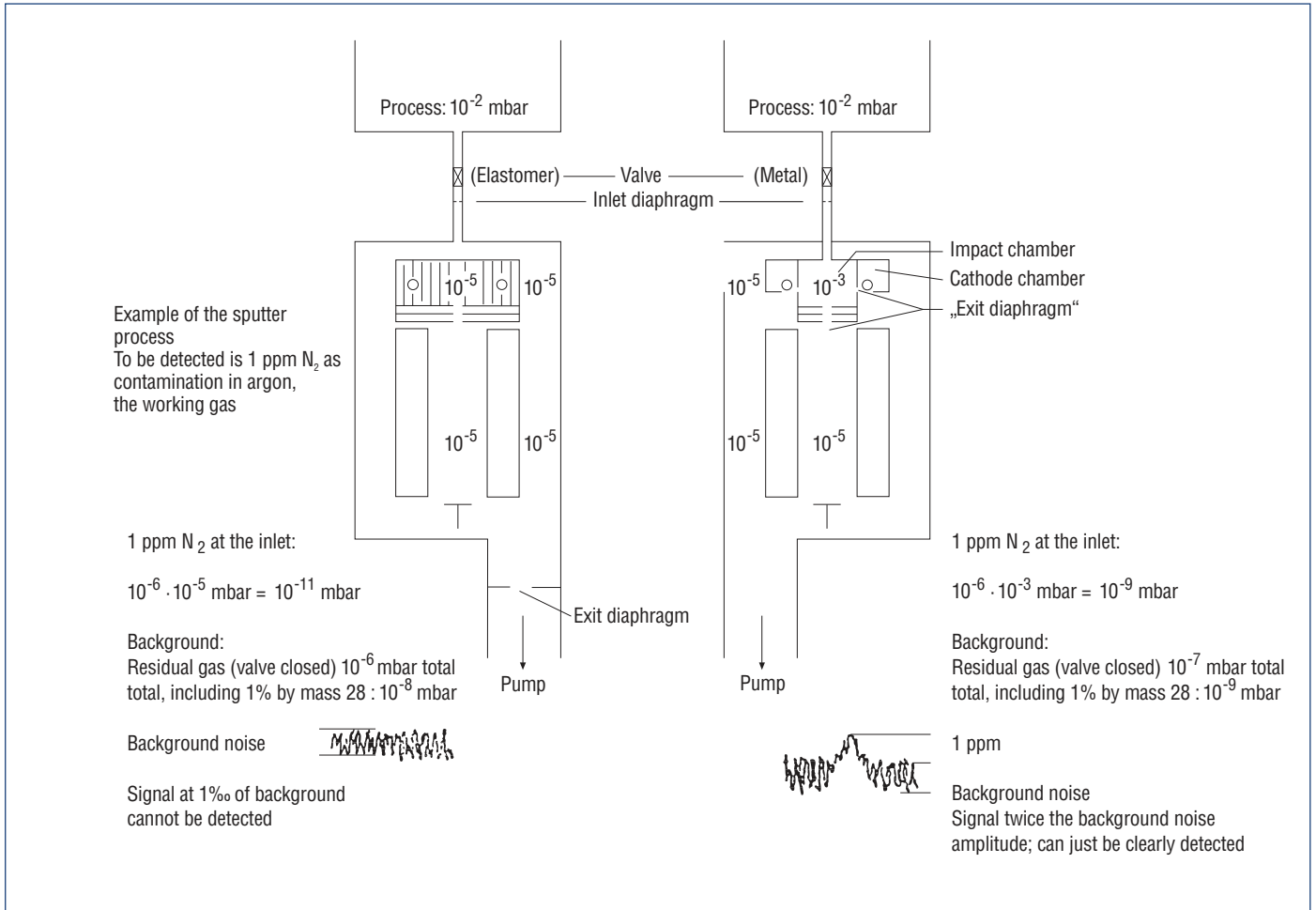


Fig. 4.8 Open ion source (left) and closed ion source (right)

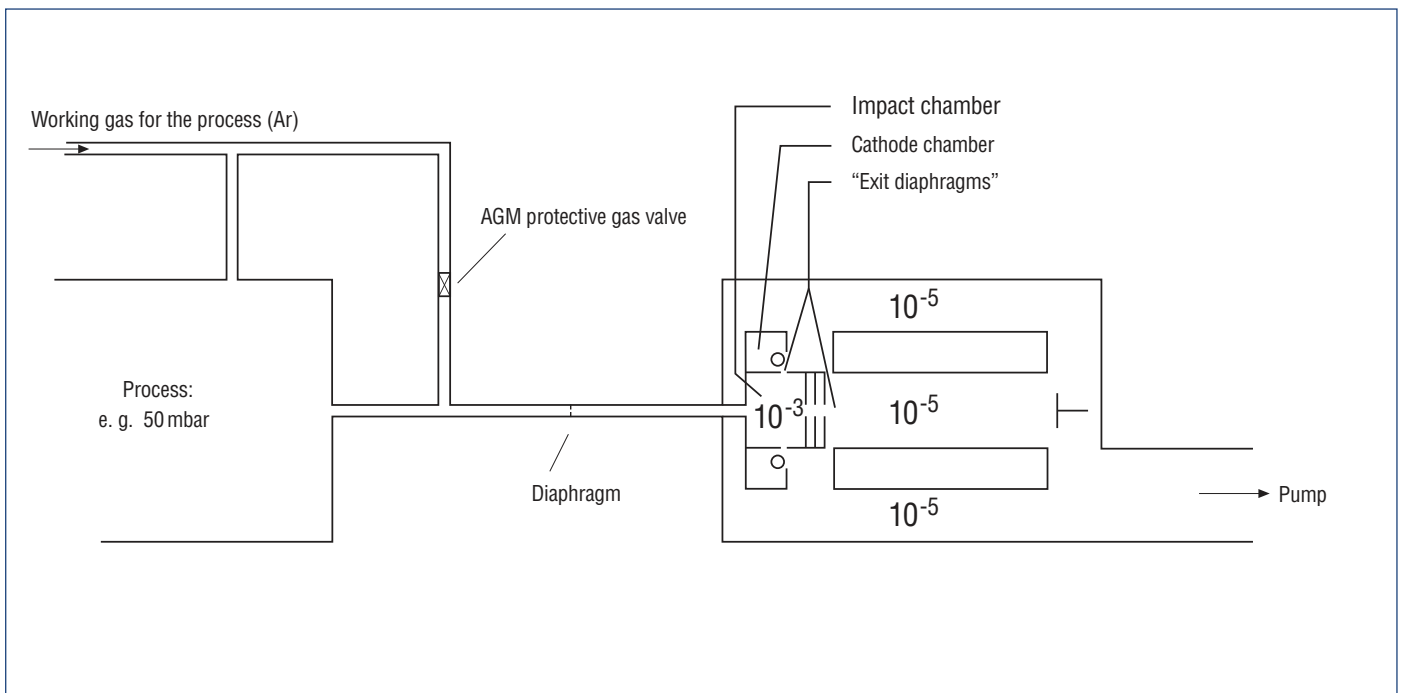


Fig. 4.9 Principle behind the aggressive gas monitor (AGM)

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: $R = M / \Delta M$ and is constant for the quadrupole spectrometer across the entire mass range, slightly greater than 1 or $\Delta M < 1$.

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 ΔM 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:

$$E = \frac{i^+}{p_G} \left(\frac{A}{\text{mbar}} \right) \quad (4.1)$$

Typical values are:

$$\text{Faraday cup: } E = 1 \cdot 10^{-4} \frac{A}{\text{mbar}}$$

$$\text{SEMP: } E = 1 \cdot 10^{+2} \frac{A}{\text{mbar}}$$

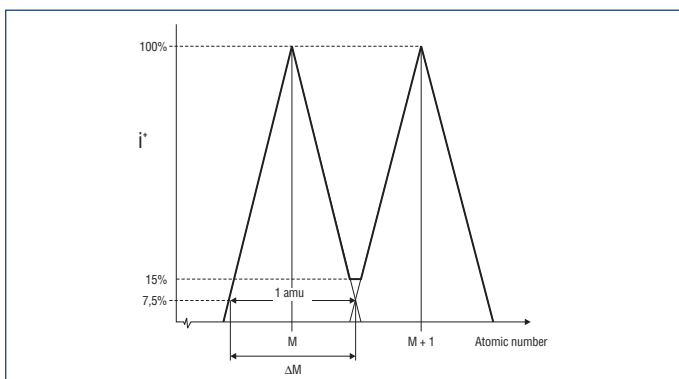


Fig. 4.10 Line width – 15 % valley

4.5.4 Smallest detectable partial pressure

The smallest detectable partial pressure is defined as a ratio of noise amplitude to sensitivity:

$$P_{\min} = \frac{\Delta \cdot i_R^+}{E} \text{ (mbar)}$$

$$\Delta \cdot i_R^+ = \text{Noise amplitude}$$

Example (from Fig. 4.11):

$$\text{Sensitivity } E = 1 \cdot 10^{-4} \frac{A}{\text{mbar}}$$

$$\text{Noise amplitude } \Delta \cdot i_R^+ = 4 \cdot 10^{-14} A$$

$$p_{\min}(\text{FC}) = \frac{4 \cdot 10^{-14} A}{1 \cdot 10^{-4} A/\text{mbar}} = 4 \cdot 10^{-10} \text{ mbar}$$

4.5.5 Smallest detectable partial pressure ratio (concentration)

The definition is:

$$\text{SDPPR} = p_{\min} / p_{\Sigma} \text{ (ppm)}$$

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:

$$0.93 \cdot 10^{-2} \cdot 0.337 \cdot 10^{-2} = 3.13 \cdot 10^{-5} = 31.3 \text{ ppm}$$

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:

$$31.3 \cdot 10^{-6} / 1.875 = 16.69 \cdot 10^{-6} = 16.69 \text{ ppm.}$$

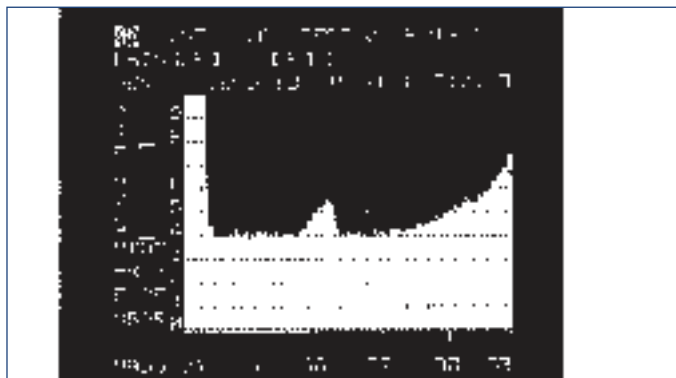


Fig. 4.11 Detection of Argon₃₆

4.5.6 Linearity range

The linearity range is that pressure range for the reference gas (N₂, Ar) in which sensitivity remains constant within limits which are to be specified (± 10 % for partial pressure measurement devices).

In the range below 1 · 10⁻⁶ mbar the relationship between the ion flow and partial pressure is strictly linear. Between 1 · 10⁻⁶ mbar and 1 · 10⁻⁴ mbar there are minor deviations from linear characteristics. Above 1 · 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 · 10⁻⁴ mbar. Depending on the information required, there will be differing upper limits for use.

In analytical applications, 1 · 10⁻⁶ mbar should not be exceeded if at all possible. The range from 1 · 10⁻⁶ mbar to 1 · 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.

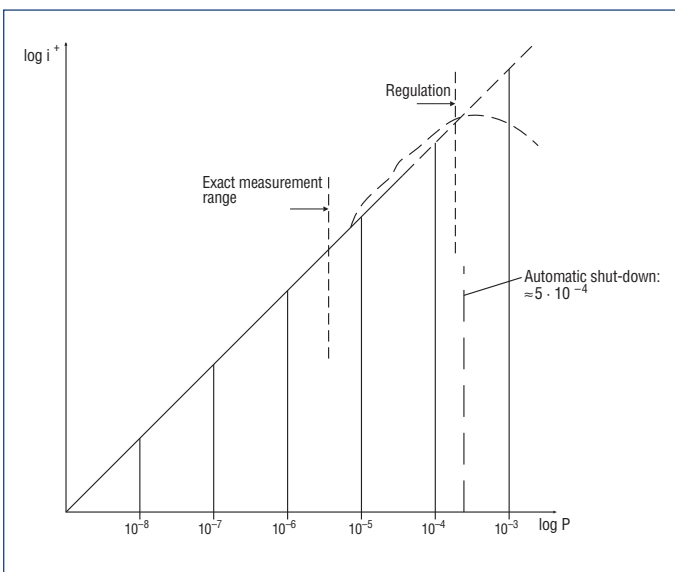


Fig. 4.12 Qualitative linearity curve

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 system ("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:

$$M = \frac{M_r}{n_e} \tag{4.2}$$

(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 CH₄⁺ and O₂⁺⁺; M = 28 for CO⁺, N₂⁺ and C₂H⁺! 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	99.985
		2	0.015
He	2	3	0.00013
		4	≈ 100.0
B	5	10	19.78
		11	80.22
C	6	12	98.892
		13	1.108
N	7	14	99.63
		15	0.37
O	8	16	99.759
		17	0.0374
		18	0.2039
F	9	19	100.0
Ne	10	20	90.92
		21	0.257
		22	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
		29	4.68
		30	3.05
P	15	31	100.0
		32	95.06
		33	0.74
		34	4.18
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

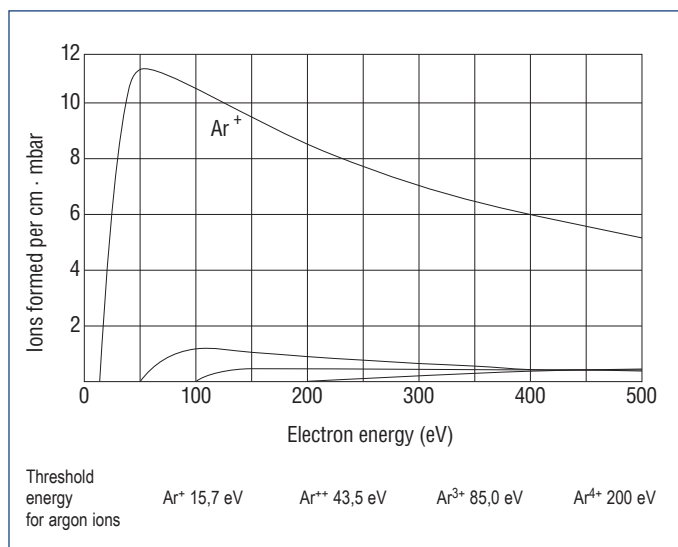


Fig. 4.13 Number of the various Ar ions produced, as a factor of electron energy level

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

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^{2+} at mass of 20 and Ar^{3+} 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).

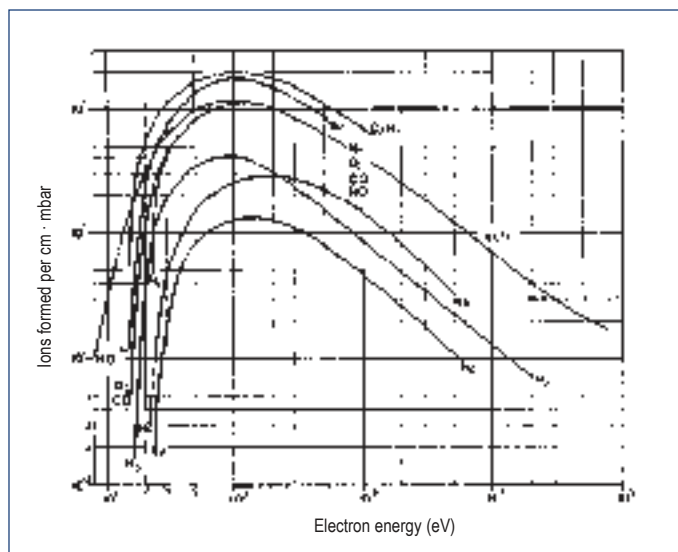


Fig. 4.14 Specific ionization S for various gases by electrons exhibiting energy level E

Type of gas	Symbol	RIP	Type of gas	Symbol	RIP
Acetone (Propanone)	(CH ₃) ₂ CO	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 ₂ S	2.2
Benzene	C ₆ H ₆	5.9	Iodine	I ₂	
Benzoic acid	C ₆ H ₅ COOH	5.5	Krypton	Kr	1.7
Bromine	Br	3.8	Lithium	Li	1.9
Butane	C ₄ H ₁₀	4.9	Methane	CH ₄	1.6
Carbon dioxide	CO ₂	1.4	Methanol	CH ₃ OH	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 ₆ H ₄ Cl	7.0	Nitrogen dioxide	N ₂ O	1.7
Chloroethane	C ₂ H ₃ Cl	4.0	Oxygen	O ₂	1.0
Chloroform	CHCl ₃	4.8	n-pentane	C ₅ H ₁₂	6.0
Chlormethane	CH ₃ Cl	3.1	Phenol	C ₆ H ₅ OH	6.2
Cyclohexene	C ₆ H ₁₀	6.4	Phosphine	PH ₃	2.6
Deuterium	D ₂	0.35	Propane	C ₃ H ₈	3.7
Dichlorodifluoromethane	CCl ₂ F ₂	2.7	Silver perchlorate	AgClO ₄	3.6
Dichloromethane	CH ₂ Cl ₂	7.8	Tin iodide	SnI ₄	6.7
Dinitrobenzene	C ₆ H ₄ (NO ₂) ₂	7.8	Sulfur dioxide	SO ₂	2.1
Ethane	C ₂ H ₆	2.6	Sulfur hexafluoride	SF ₆	2.3
Ethanol	C ₂ H ₅ OH	3.6	Toluene	C ₆ H ₅ CH ₃	6.8
Ethylene oxide	(CH ₂) ₂ O	2.5	Trinitrobenzene	C ₆ H ₃ (NO ₂) ₃	9.0
Helium	He	0.14	Water vapor	H ₂ O	11.0
Hexane	C ₆ H ₁₄	6.6	Xenon	Xe	3.0
Hydrogen	H ₂	0.44	Xylols	C ₆ H 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
Neon	Ne	12	3.5	3.8	4.6	5
		22	9.2	10.2	0.9	11
		20	89.6	100	90.1	100
Oxygen	O ₂	10	0.84	0.93	9	4
		34	0.45	0.53		
		32	84.2	100	90.1	100
Nitrogen	N ₂	16	15.0	17.8	9.9	11
		29	0.7	0.8	0.9	1
		28	86.3	100	92.6	100
Water vapor	H ₂ O	14	12.8	15	6.5	12
		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) ₂ CO	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 ₆ H 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 ₃ CH ₂ OH	31/100	45/34	27/24	29/23	46/17	26/8
14	Halocarbon 11	CCl ₃ F	101/100	103/60	35/16	66/15	47/12	31/10
15	Halocarbon 12	CCl ₂ F 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 ₂ C ₁₃ F ₃	101/100	103/62	85/55	31/50	151/41	153/25
20	Helium	He	4/100	-	-	-	-	-
21	Heptane	C ₇ H ₁₆	43/100	41/62	29/49	27/40	57/34	71/28
22	Hexane	C ₆ H 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 ₂ S	34/100	32/44	33/42	36/4	35/2	-
25	Isopropyl alcohol	C ₃ H ₈ O	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	Methyl alcohol	CH ₃ OH	31/100	29/74	32/67	15/50	28/16	2/16
29	Methyl ethyl ketone	C ₄ H ₈ O	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 ₆ H ₅ CH ₃	91/100	92/62	39/12	65/6	45.5/4	51/4
39	Trichloroethane	C ₂ H ₃ Cl ₃	97/100	61/87	99/61	26/43	27/31	63/27
40	Trichloroethylene	C ₂ HCl 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 ₂ O	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

of the share of N₂⁺ with its mass of 28. If, on the other hand, no oxygen is 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 CO₂ (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 (N₂⁺⁺) will always be found in the spectrum in addition to the atomic number 28 (N₂⁺); 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 (O₂⁺⁺).

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.

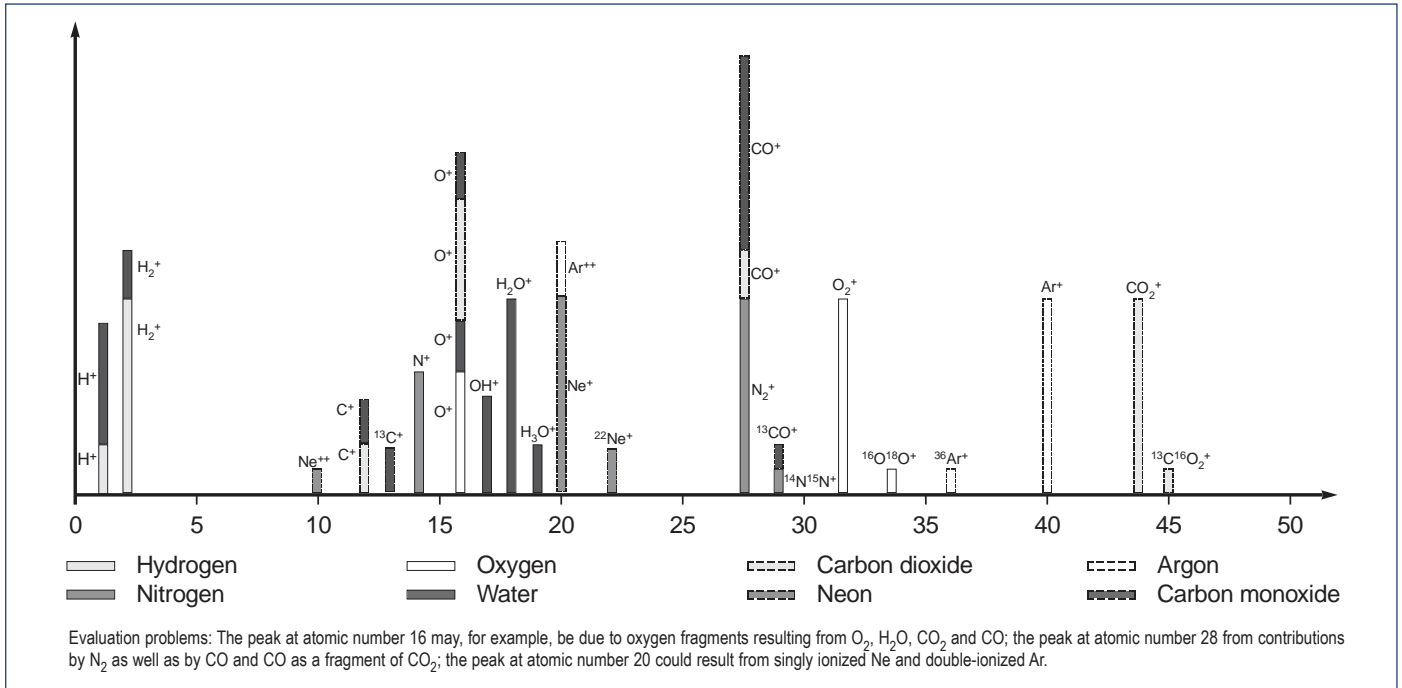


Fig. 4.15 Model spectrum

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 E_{gas} times the partial pressure p_{gas} :

$$i_{gas}^+ (\text{produced}) = i^- \cdot S_{gas} \cdot f \cdot RIP_{gas} \cdot p_{gas}$$

$$= E_{gas} \cdot p_{gas}$$

$$\text{due to } RIP_{N_2} = 1$$

$$EN_2 \text{ is equal to } i^- \cdot SN_2 \cdot f$$

$$\text{and } E_{gas} \text{ is equal to } EN_2 \cdot RIP_{gas}$$

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:

$$\begin{aligned} i_{gas}^+ (\text{produced}) &= i_{gas,m_1}^+ + i_{gas,m_2}^+ + \dots = \frac{i_{gas,m_1}^+}{BF_{gas,m_1}} \\ &= \frac{i_{gas,m_2}^+}{BF_{gas,m_2}} = \dots = E_{gas} \cdot p_{gas} \end{aligned}$$

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

$$\begin{aligned} p_{gas} &= \frac{i_{gas,m_2}^+ (\text{produced})}{BF_{gas,m_2} \cdot E_{gas}} \\ \Rightarrow p_{gas} &= \frac{i_{gas,m_2}^+ (\text{measured})}{BF_{gas,m_2} \cdot E_{gas} \cdot TF(m)} \end{aligned}$$

and with

$$a \quad E_{gas} = EN_2 \cdot RIP_{gas}$$

the ultimate result is:

$$p_{gas} = i_{gas,m_2}^+ (\text{measured}) \cdot \frac{1}{EN_2} \cdot \frac{1}{BF_{gas,m_2} \cdot RIP_{gas} \cdot TF(m)} \quad (4.3)$$

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.

- 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.

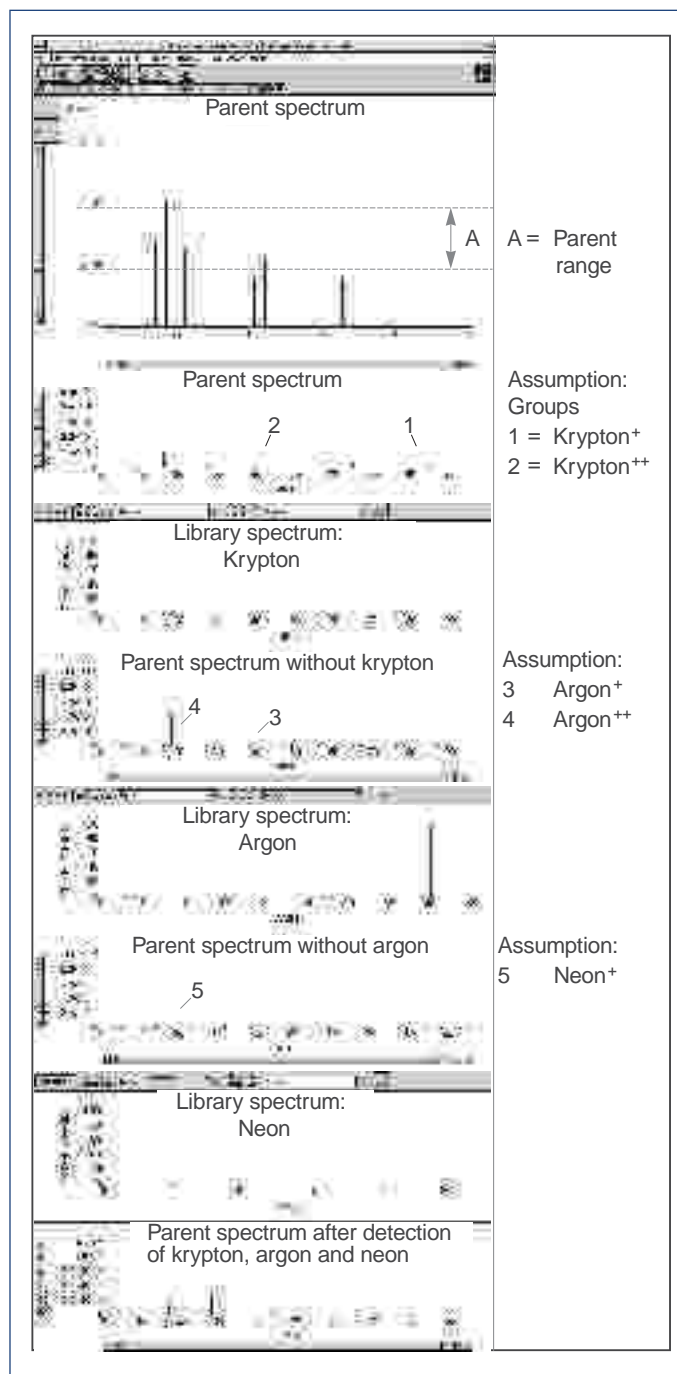


Fig. 4.16 Subtracting spectra contained in libraries

In the most general case a plurality of gases will make a greater or lesser 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 $F_{m,g}$. In order to simplify calculation, the fragment factor $F_{m,g}$ 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:

$$\begin{bmatrix} i_j^+ \\ \vdots \\ i_m^+ \\ \vdots \\ i_u^+ \end{bmatrix} = \begin{bmatrix} BF_{j,k} & \dots & \dots & \dots & BF_{j,o} \\ \vdots & \vdots & \vdots & \vdots & \vdots \\ \vdots & \vdots & FF_{m,g} & \vdots & \vdots \\ \vdots & \vdots & \vdots & \vdots & \vdots \\ BF_{u,k} & \dots & \dots & \dots & BF_{u,o} \end{bmatrix} \begin{bmatrix} I_k^+ \\ \vdots \\ I_g^+ \\ \vdots \\ I_o^+ \end{bmatrix}$$

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

$$\sum_{g=k}^0 BF_{m,g} = \text{fragment matrix}$$

I_g^+ = Vector of the sum of the flows for the individual gases or:

$$i_m^+ = \sum p_g \cdot E_{N_2} \cdot \overbrace{RIP_g \cdot FF_m \cdot TF_m}^{F_{m,g}}$$

$F_{m,g}$
 Fragment factor for the gas to mass m
 Relative ionization probability for the gas
 Nitrogen sensitivity (equipment constant)
 Partial pressure of the gas
 Ion current for atomic number m

Transmission factor for the mass m
 Fragment factor for the gas to mass m
 Relative ionization probability for the gas
 Nitrogen sensitivity (equipment constant)
 Partial pressure of the gas
 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 SOX software (DOS) for stand-alone operation (1 MS plus 1 PC, RS 232)

The conventional software package (SOX) 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 MOX (1 to 8 MS plus 1 PC, RS 485)

The first step toward process-oriented software by LEYBOLD is the MOX. It makes possible simultaneous monitoring of a maximum of eight sensors and you can apply all the SOX 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

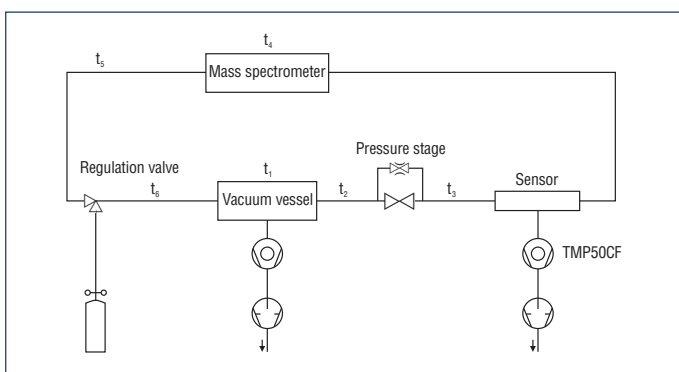


Fig. 4.17 Partial shares for overall time constants

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 $7.2 \cdot 10^{-6}$ mbar to $7.2 \cdot 10^{-5}$ mbar (or by a factor of just 10 overall).

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 $1 \cdot 10^{-4}$ to $1 \cdot 10^{-3}$ 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.

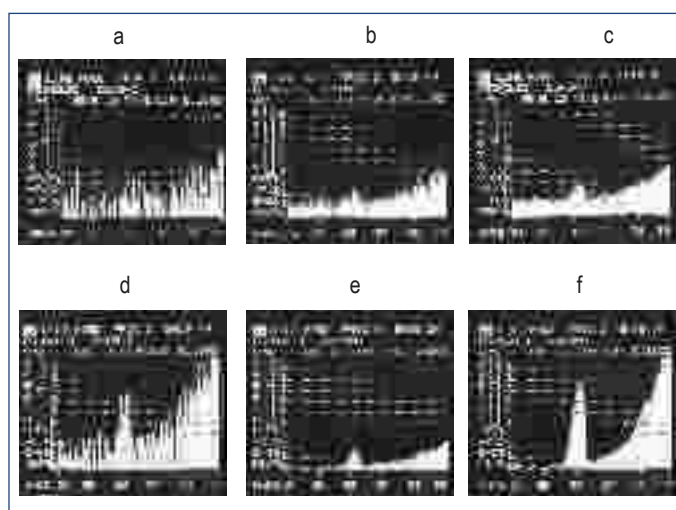


Fig. 4.18 Improving the signal-to-noise ratio by increasing the pressure or extending the integration time

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 $\text{mbar} \cdot \text{l/s}$ or cm^3/s (STP) as the unit of measure. A leak rate of $Q_L = 1 \text{ mbar} \cdot \text{l/s}$ 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 $\text{mbar} \cdot \text{l/s}$. 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:

$$Q_L = \frac{\Delta(p \cdot V)}{\Delta t} = \frac{R \cdot T}{M} \cdot \frac{\Delta m}{\Delta t} \quad (5.1)$$

where $R = 83.14 \text{ mbar} \cdot \text{l/mol} \cdot \text{K}$, 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 $M(\text{R12}) = 121 \text{ g/mole}$:

$$\begin{aligned} Q_L &= \frac{\Delta(p \cdot V)}{\Delta t} = \frac{83.14 \text{ mbar} \cdot \ell \cdot 298 \text{ K} \cdot 1 \text{ g}}{\text{mol} \cdot \text{K} \cdot 121 \text{ g} \cdot \text{mol}^{-1} \cdot 1 \text{ year}} \\ &= \frac{83.14 \cdot 2.98 \cdot 10^2 \cdot 1}{121 \cdot 1} \cdot \frac{\text{mbar} \cdot \ell}{3.15 \cdot 10^7 \text{ s}} \\ &= \frac{83.14 \cdot 2.98 \cdot 10^2}{1.21 \cdot 10^2 \cdot 3.15} \cdot 10^{-7} \cdot \frac{\text{mbar} \cdot \ell}{\text{s}} \\ &= 65 \cdot 10^{-7} \cdot \frac{\text{mbar} \cdot \ell}{\text{s}} \end{aligned}$$

Thus the Freon loss comes to $Q_L = 6.5 \cdot 10^{-6} \text{ mbar} \cdot \text{l/s}$. 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 VIIIb in Chapter 9.

The following rule of thumb for quantitative characterization of high vacuum equipment may be applied:

Total leak rate $< 10^{-6} \text{ mbar} \cdot \text{l/s}$:
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):

$$p_{end} = \frac{Q_L}{S_{eff}} \quad (5.2)$$

(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

$\Delta p = 1013$ mbar, Hole diameter $d = 1$ cm	
Gas speed = Speed of sound = $330 \frac{m}{s}$	
Volume/second:	$330 \frac{m}{s} \cdot \frac{1^2 \cdot \pi}{4} \text{ cm}^2 = 25.95 \cdot 10^{-3} \frac{\text{cm}^3}{s} = 25.95 \frac{\ell}{s}$
Quantity/second:	$1013 \text{ mbar} \cdot 25.95 \frac{\ell}{s} = 2.63 \cdot 10^{14} \approx 10^{14} \frac{\text{mbar} \cdot \ell}{s}$
Diameter cm	Leak rate $\frac{\text{mbar} \cdot \ell}{s}$
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

hole 1 cm in diameter Q_L (air) will be $2.6 \cdot 10^{14}$ mbar · 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 $7.7 \cdot 10^{14}$ mbar · 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).

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^{14} mbar · 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 · 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 · 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 · 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. $\approx 1 \mu\text{m}$
(rod-shaped)			Avg. $\approx 0.5\text{-}1 \mu\text{m}$, 2–10 μm long
Oil-tight		$Q_L < 10^{-5}$	
Virus-tight*)			
(vaccines such as pox)		$Q_L < 10^{-6}$	$\varnothing \approx 3 \cdot 10^{-7}$ m
(smallest viruses, bacteriophages)		$Q_L < 10^{-8}$	$\varnothing \approx 3 \cdot 10^{-8}$ m
(viroids, RNA)		$Q_L < 10^{-10}$	$\varnothing \approx 1 \cdot 10^{-9}$ 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

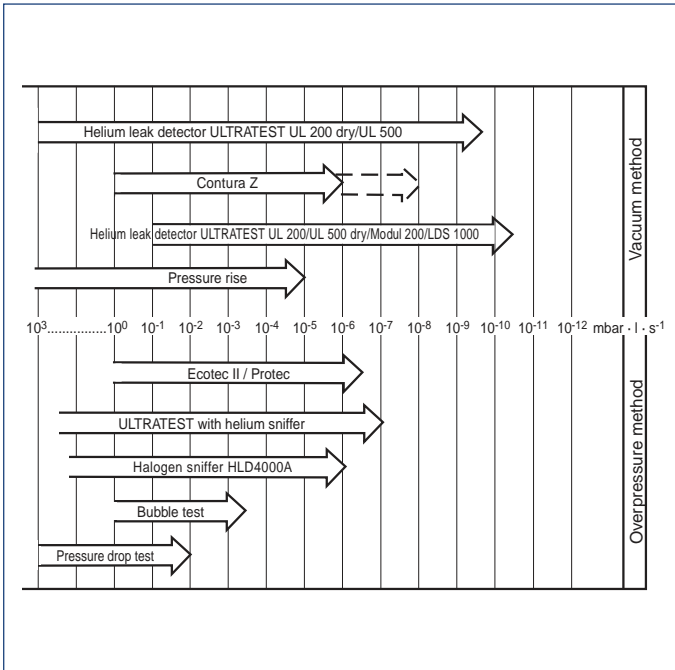


Fig. 5.2 Leak rate ranges for various leak detection processes and devices

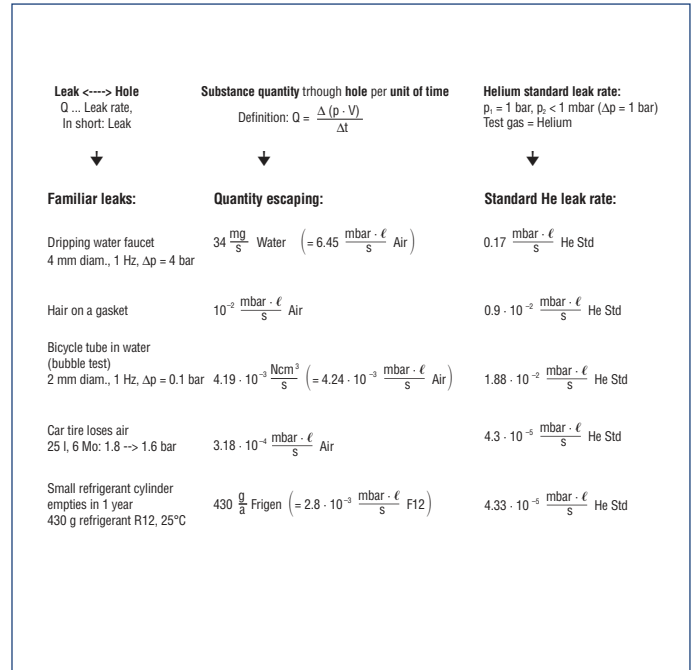


Fig. 5.3 Examples for conversion into helium standard leak rates

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	$Q_I \cdot (p_1^2 - p_2^2)_{II} = Q_{II} \cdot (p_1^2 - p_2^2)_I$	$Q_I \cdot (p_1 - p_2)_{II} = Q_{II} \cdot (p_1 - p_2)_I$
Gas	$Q_{gas A} \cdot \eta_{gas A} = Q_{gas B} \cdot \eta_{gas B}$	$Q_{gas A} \cdot \sqrt{M_{gas A}} = Q_{gas B} \cdot \sqrt{M_{gas B}}$

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.

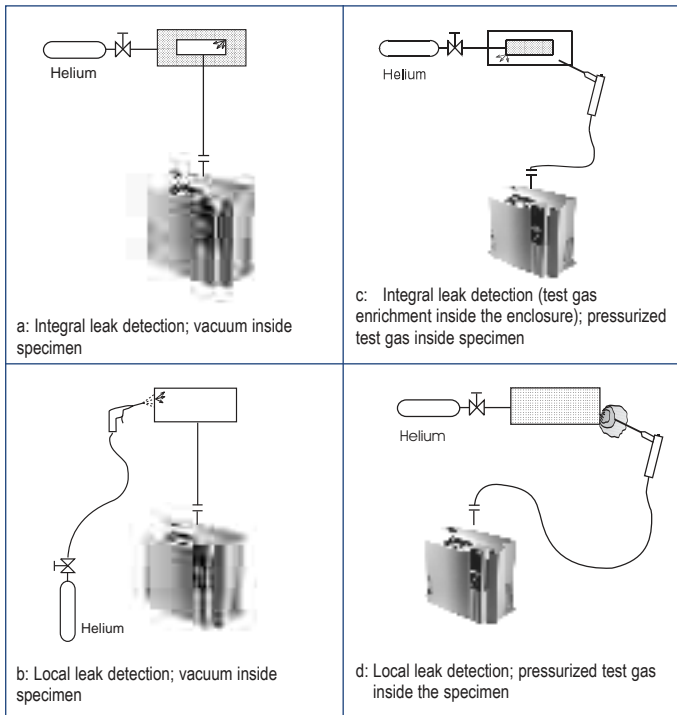


Fig. 5.4 Leak test techniques and terminology

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 as 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 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

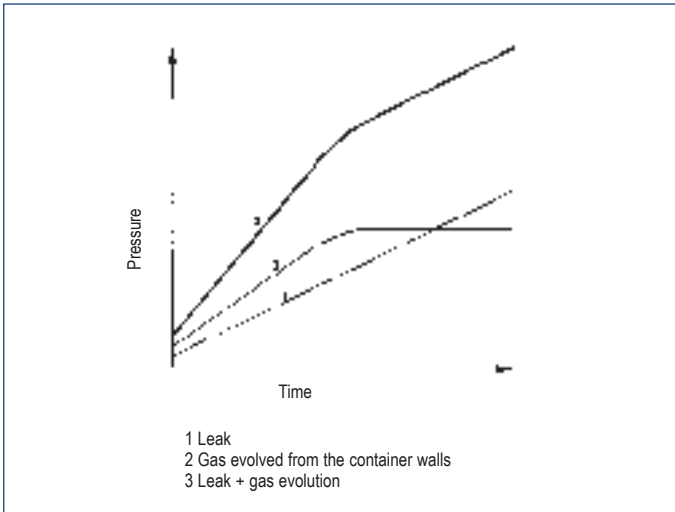


Fig. 5.5 Pressure rise within a vessel after the pump is switched off

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 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:

$$Q_L = \frac{\Delta p \cdot V}{\Delta t} \quad (5.3)$$

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

$$Q_L = \frac{(1 \cdot 10^{-3} - 1 \cdot 10^{-4}) \cdot 20}{300} = \frac{9 \cdot 10^{-4} \cdot 20}{300} = 6 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}}$$

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

$$Q_L = \frac{\Delta m}{\Delta t} = 6 \cdot 10^{-5} \frac{\text{mbar} \cdot \ell}{\text{s}} \cdot \frac{29 \frac{\text{g}}{\text{mol}}}{83.14 \text{ mbar} \cdot \ell \cdot 293 \cdot 10^2 \text{ K}} = 7 \cdot 10^{-8} \frac{\text{g}}{\text{s}}$$

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 $S_{\text{eff}} = 30$ l/s. Thus the ultimate pressure will be

$$p_{\text{end}} = \frac{Q_L}{S_{\text{eff}}} = \frac{6 \cdot 10^{-5} \text{ mbar} \cdot \ell \cdot \text{s}^{-1}}{30 \ell \cdot \text{s}^{-1}} = 2 \cdot 10^{-6} \text{ mbar}$$

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 ionizability 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 · 10 ⁻³	a few seconds
84	40	5.4 · 10 ⁻⁴	a few seconds
28	145	1.8 · 10 ⁻⁴	a few seconds
14	290	9.0 · 10 ⁻⁵	a few seconds
2.8	24 min	1.8 · 10 ⁻⁵	a few seconds
0.28 *	6 h	1.8 · 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) with helium leak

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. 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}	$7 \cdot 10^{-3}$	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 · l/s for all the devices. Equipment operating in accordance with the halogen diode principle can also detect SF₆. Consequently these sniffer units are used to determine whether refrigerants are escaping from a refrigeration unit or from an SF₆ 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 · 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:

- 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
- 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 possible 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 through 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:

$$Q = p \cdot S \tag{5.4}$$

At all other points

$$Q = p \cdot S_{\text{eff}} \tag{5.4a}$$

applies, taking the line losses into account.

The equation applies to all gases which are pumped through the piping and thus also for helium.

$$Q_{\text{He}} = p_{\text{He}} \cdot S_{\text{eff, He}} \tag{5.4b}$$

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

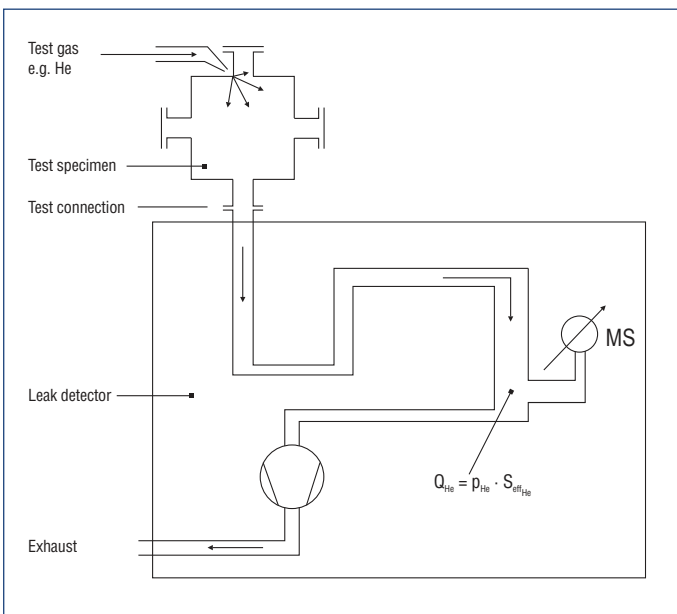


Fig. 5.6 Basic operating principle for a leak detector

microprocessor to multiply the signal arriving from the mass spectrometer by a numerical constant and to have the **leak rate displayed direct**.

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

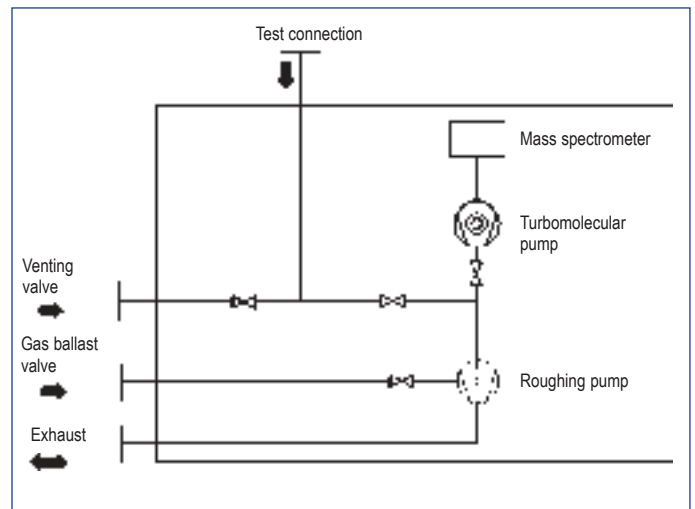


Fig. 5.7 Correct set-up for a MSLD

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.

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 5.7.1) 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.

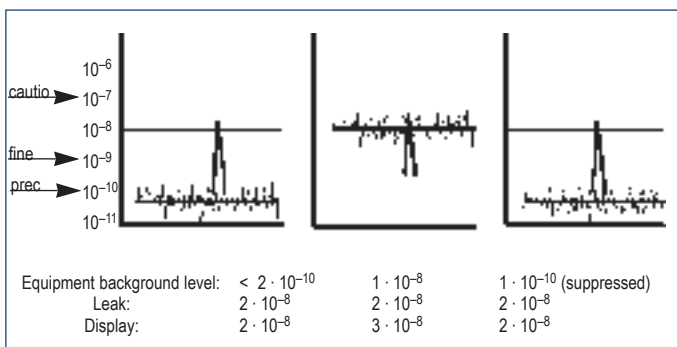


Fig. 5.8 Example of zero-point suppression

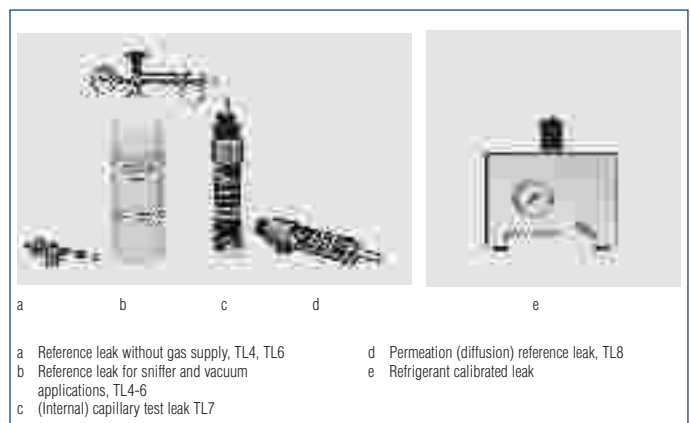


Fig. 5.9 Examples for the construction of test leaks

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.

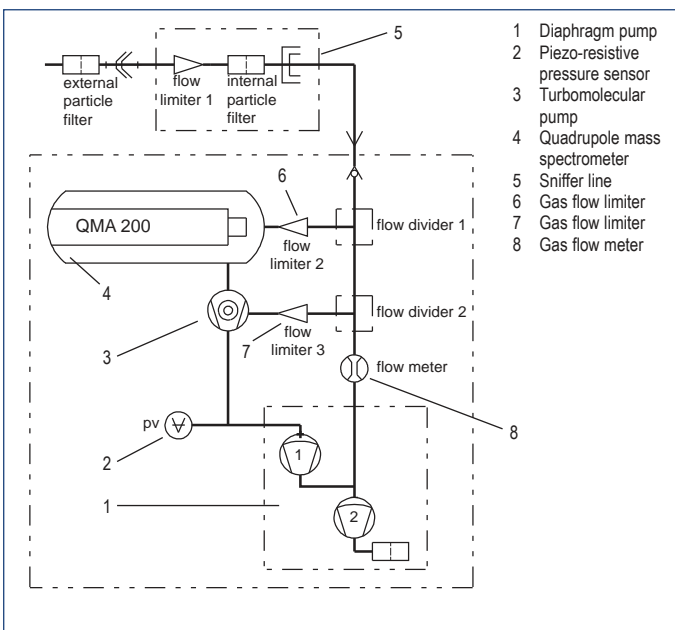


Fig. 5.10 Vacuum schematic for the ECOTEC II

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 through 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

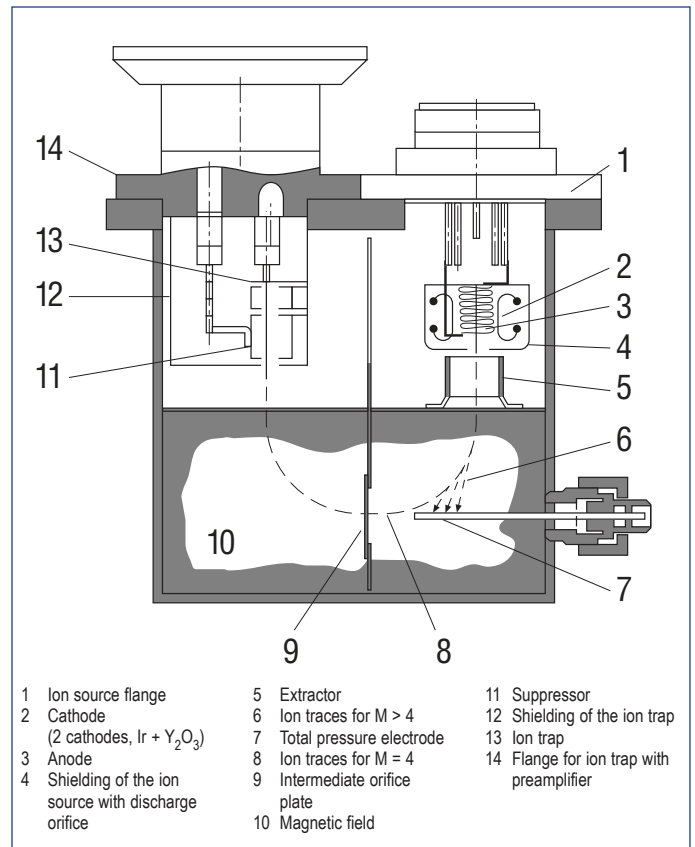


Fig. 5.11 Configuration of the 180° sector mass spectrometer

evaluated 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 S_{eff} used in equation 5.4b is the pumping speed of the turbomolecular pump at the ion source location:

$$Q_{\text{He}} = p_{\text{He}} \cdot S_{\text{eff, turbomolecular pump ion source}} \quad (5.5a)$$

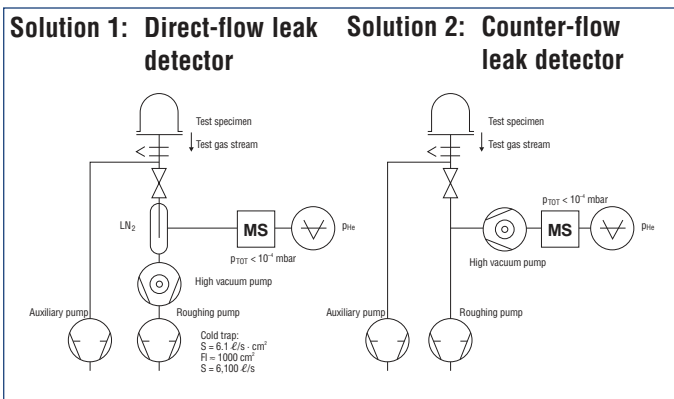


Fig. 5.12 Full-flow and counter-flow leak detector

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 $p_{\text{min, He}} = 1 \cdot 10^{-12}$ mbar. The pumping speed for helium would be $S_{\text{He}} = 10$ l/s. Then the smallest detectable leak rate is $Q_{\text{min}} = 1 \cdot 10^{-12}$ mbar · 10 l/s = $1 \cdot 10^{-11}$ mbar · 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 · 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:

$$Q_{\text{He}} = p_{\text{He}} \cdot S_{\text{eff}} \cdot K \quad (5.5b)$$

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:

$$Q_{\text{Vacuum vessel}} = \gamma \cdot \text{Display}_{\text{Leak detector}} \quad (5.6)$$

where γ 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, γ can be determined with a reference leak attached at the vacuum vessel:

$$\gamma = \frac{\text{Display at the leak detector}}{Q_L \text{ for the reference leak}} \quad (5.7)$$

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

Partial flow principle (example)

$V = 150 \text{ l}$
 $Q_{\text{He}} = 3 \cdot 10^{-5} \frac{\text{mbar} \cdot \text{l}}{\text{s}}$ (Leak rate)

$S_{\text{eff}} = S_{\text{PFP}} + S_{\text{LD}} \rightarrow S_{\text{LD}} = 8 \frac{\text{l}}{\text{s}}$ Leak detector (LD)
 $S_{\text{PFP}} = 60 \frac{\text{m}^3}{\text{s}} = 16.66 \frac{\text{l}}{\text{s}}$ Partial flow pump (PFP)

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
 Overall pumping speed: $S_{\text{eff}} = S_{\text{LD}} + S_{\text{PFP}} = 8 + 16.66 = 24.66 \frac{\text{l}}{\text{s}}$

γ ... Partial flow ratio

Signal to Leak detector: $3 \cdot 10^{-5} \frac{\text{mbar} \cdot \text{l}}{\text{s}} \cdot \frac{8 \frac{\text{l}}{\text{s}}}{(8 + 16.66) \frac{\text{l}}{\text{s}}} = 9.73 \cdot 10^{-6} \frac{\text{mbar} \cdot \text{l}}{\text{s}}$
 Signal to partial flow pump: $3 \cdot 10^{-5} \frac{\text{mbar} \cdot \text{l}}{\text{s}} \cdot \frac{16.66 \frac{\text{l}}{\text{s}}}{(8 + 16.66) \frac{\text{l}}{\text{s}}} = 2.02 \cdot 10^{-5} \frac{\text{mbar} \cdot \text{l}}{\text{s}}$

Check: Overall signal $Q_{\text{He}} = Q_{\text{LD}} + Q_{\text{PFP}} = 3.00 \cdot 10^{-5} \frac{\text{mbar} \cdot \text{l}}{\text{s}}$

Partial flow ratio = Fraction of the overall flow to the leak detector

$$\gamma = \frac{Q_{\text{LD}}}{Q_{\text{He}}} = \frac{Q_{\text{LD}}}{Q_{\text{LD}} + Q_{\text{PFP}}} = \frac{1}{1 + \frac{Q_{\text{PFP}}}{Q_{\text{LD}}}}$$

$$Q_{\text{LD}} = \gamma \cdot Q_{\text{He}}$$

or $\gamma = \frac{S_{\text{LD}}}{S_{\text{LD}} + S_{\text{PFP}}} = \frac{1}{1 + \frac{S_{\text{PFP}}}{S_{\text{LD}}}}$

B) Response time: $t_{95\%} = 3 \cdot \frac{V}{S_{\text{eff}}} = 3 \cdot \frac{150 \text{ l}}{24.66 \frac{\text{l}}{\text{s}}} = 18.25 \text{ s}$
 Estimate: Value for S, V and γ are uncertain \rightarrow certain: calibrate with reference leak

Fig. 5.13 Partial flow principle

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.

5.5.2.9 Time constants

The time constant for a vacuum system is set by

$$\tau = \frac{V}{S_{\text{eff}}} \quad (5.8)$$

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:

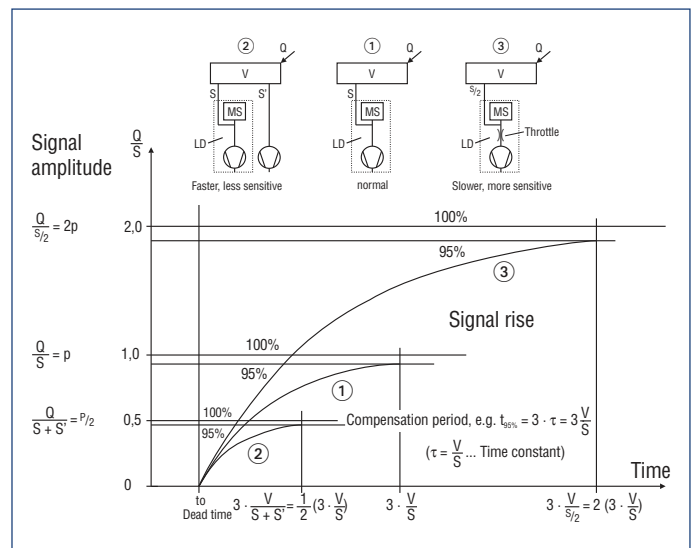


Fig. 5.14 Signal responses and pumping speed

- Center: The specimen with volume of V is joined directly with the leak detector LD (effective pumping speed of S).
- Left: In addition to 1, a partial flow pump with the same effective pumping speed, $S^1 = S$, is attached to the test specimen.
- Right: As at 1, but S is throttled down to $0.5 \diamond S$.

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:

$$p_{He} = \frac{Q}{S_{eff}} \cdot \left(1 - e^{-\frac{t}{\tau}}\right) \quad (5.9)$$

The signal will attain a prortion of its ultimate value after

$t = 1 \tau \dots 63.3 \%$	$t = 2 \tau \dots 86.5 \%$
$t = 3 \tau \dots 95.0 \%$	$t = 4 \tau \dots 98.2 \%$
$t = 5 \tau \dots 99.3 \%$	$t = 6 \tau \dots 99.8 \%$

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

- The **smallest detectable leak rate**.
- The **effective pumping speed at the test connection**.
- 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**:

$$Q_{max} = p_{max} \cdot S_{eff, connector} \quad (5.10)$$

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.

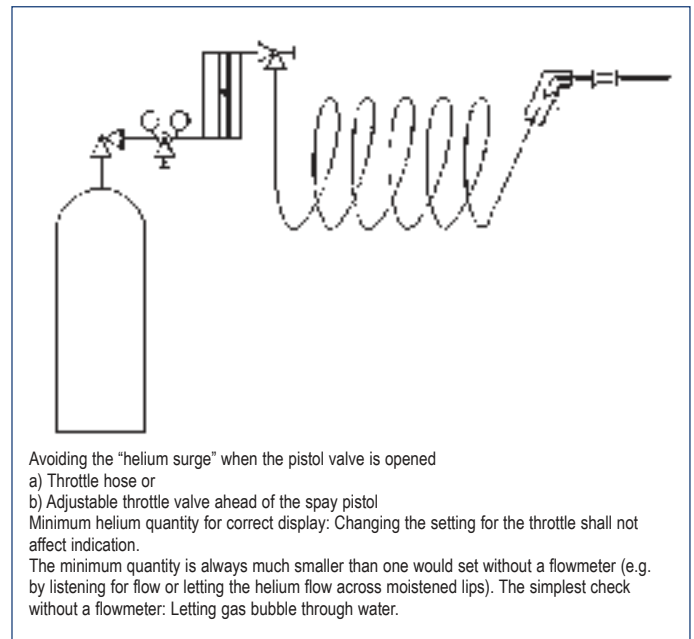
5.7 Leak detection techniques using helium leak detectors

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 5



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

ppm share of helium in the air is sufficient for detection purposes. The leak rate is then:

$$\frac{\text{Display (pure He)}}{1} = \frac{\text{Display (atmosph. He)}}{5 \cdot 10^{-6}}$$

$$Q_L = \text{Display (pure He)} \quad (5.11)$$

$$= 2 \cdot 10^{+5} \cdot \text{Display (atmospheric He)}$$

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 used 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 $35^\circ 10'$ 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.

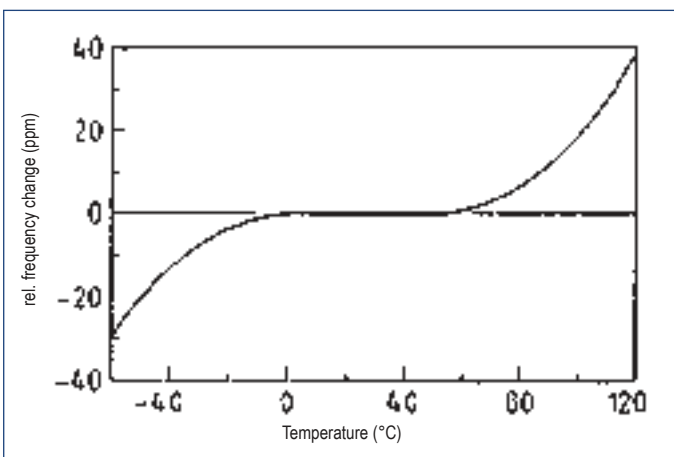


Figure 6.1 Natural frequency as a function of temperature in an AT cut quartz crystal

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:

$$\frac{M_f}{M_q} = \frac{\Delta F}{F_q} \quad \text{or} \quad M_f = M_q \cdot \frac{\Delta F}{F_q} \quad \text{with} \quad (6.1)$$

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 $M_q = D_q \cdot \rho_q \cdot A$, 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:

$$D_f = \frac{F_q}{F_c} \cdot D_q \cdot \rho_q \cdot \frac{\Delta F}{F_q \cdot \rho_f} = K \cdot \frac{\Delta F}{\rho_f} \quad \text{with}$$

$$K = \frac{D_q \cdot F_q \cdot \rho_q}{F_q^2} = \frac{N_{AT} \cdot \rho_q}{F_q^2} \quad \text{where}$$

$N = F_q \cdot D_q$ is the frequency constant (for the AT cut $N_{AT} = 166100 \text{ Hz} \cdot \text{cm}$) and $\rho_q = 2.649 \text{ g/cm}^3$ is the density of the quartz. The coating thickness is thus proportional to the frequency shift ΔF and inversely proportional to the

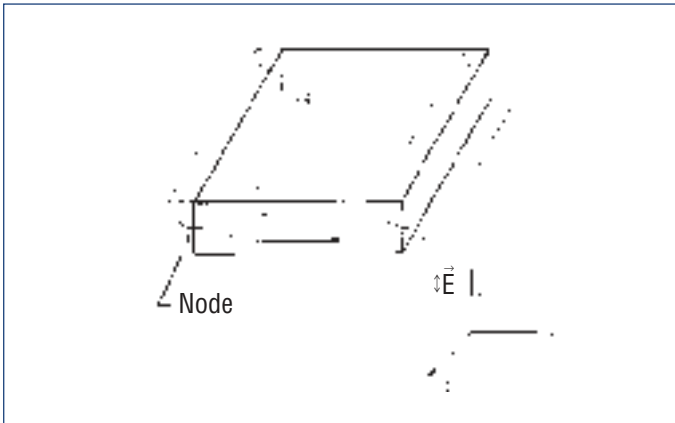


Fig. 6.2 Thickness shear oscillations

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 1 Å of aluminum ($d_e = 2.77 \text{ g/cm}^3$). 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

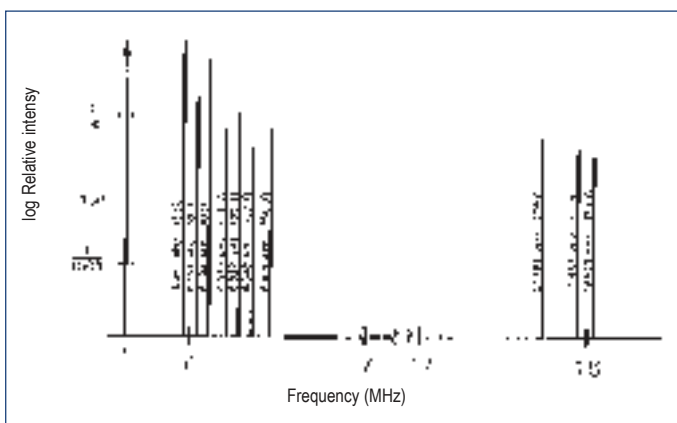


Fig. 6.4 Frequency resonance spectrum

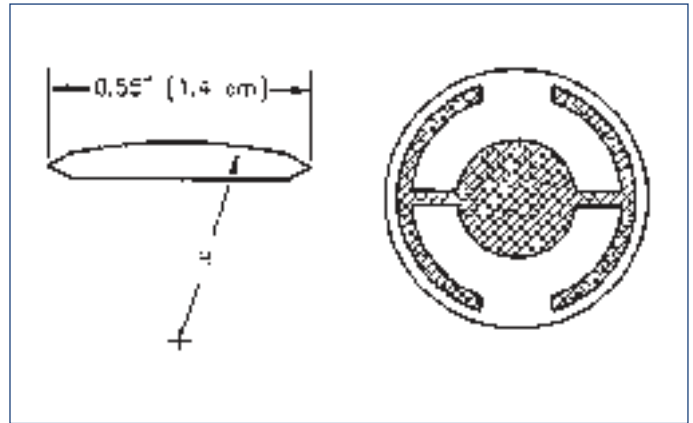


Fig. 6.3 Shape of LEYBOLD-Inficon quartz crystals

oscillation, which is called the fundamental wave. The characteristic 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 Behrmdt discovered that:

$$\frac{M_f}{M_q} = \frac{(T_c - T_q)}{T_q} = \frac{\Delta F}{F_c} \quad \text{with (6.3)}$$

$T_c = 1 / F_c$... 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:

$$T_f = \left(\frac{N_{AT} \cdot d_q}{\pi \cdot d_f \cdot F_c \cdot Z} \right) \cdot \arctg \left(Z \cdot \tg \left[\frac{\pi \cdot (F_q - F_c)}{F_q} \right] \right) \quad (6.4)$$

$$Z = \frac{\sqrt{d_q \cdot U_q}}{d_f \cdot U_f} \quad \text{acoustic impedance ratio}$$

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.

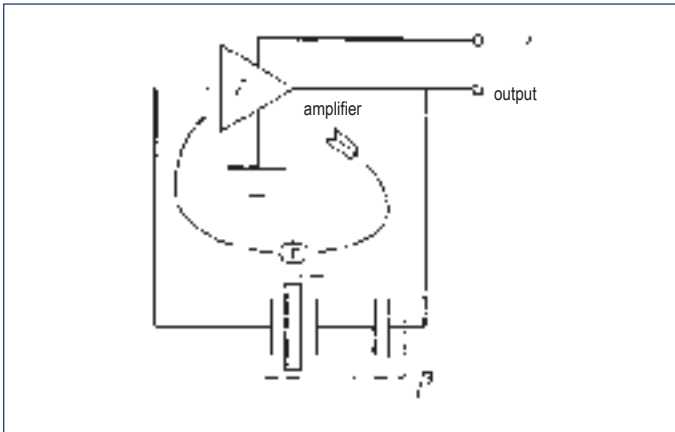


Fig. 6.5 Circuit of the active oscillator

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

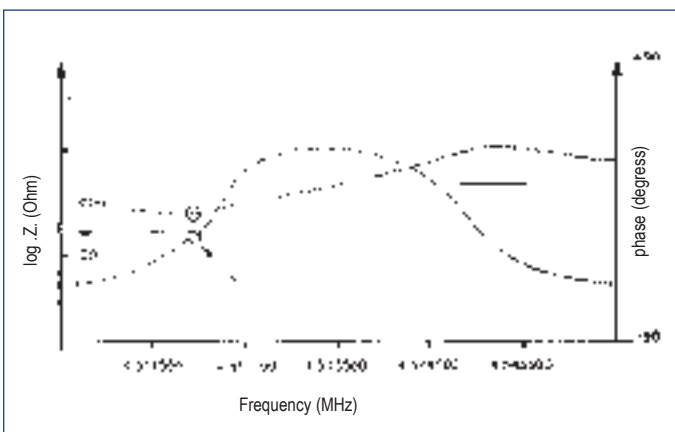


Fig. 6.7 Oscillations of a thickly coated crystal

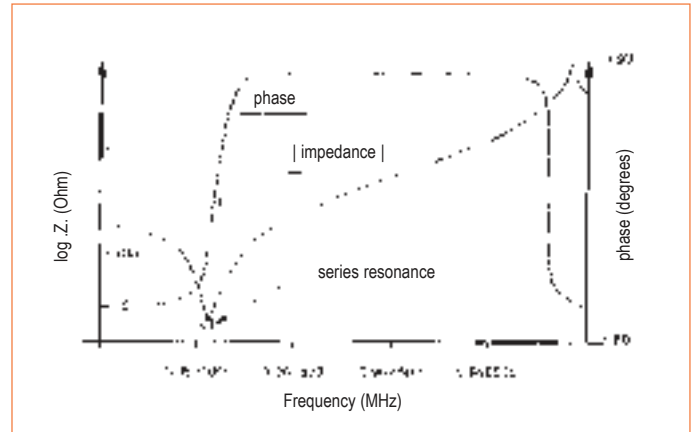


Fig. 6.6 Crystal frequencies near the series resonance point

suddenly significantly smaller, i.e. by the amount of the frequency difference between the fundamental wave and the anharmonic adopted by the oscillation.

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:

$$\frac{(C_{55}/C_{66})_{\text{coated}}}{(C_{55}/C_{66})_{\text{uncoated}}} \approx \frac{1}{(1 + M \cdot Z)} \quad (6.5)$$

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 $M \pi Z$ also appears in the Lu-Lewis equation (equation 6.4). It can be used to assess the effective Z value from the following equations:

$$\text{tg} \left(M \cdot Z \cdot \pi \cdot \frac{F_c}{F_q} \right) + Z \cdot \text{tg} \left(\pi \cdot \frac{F_c}{F_q} \right) = 0 \quad (6.6)$$

$$\text{or } Z = - \frac{\text{tg} \left(M \cdot Z \cdot \pi \cdot \frac{F_c}{F_q} \right)}{\text{tg} \left(\pi \cdot \frac{F_c}{F_q} \right)}$$

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.

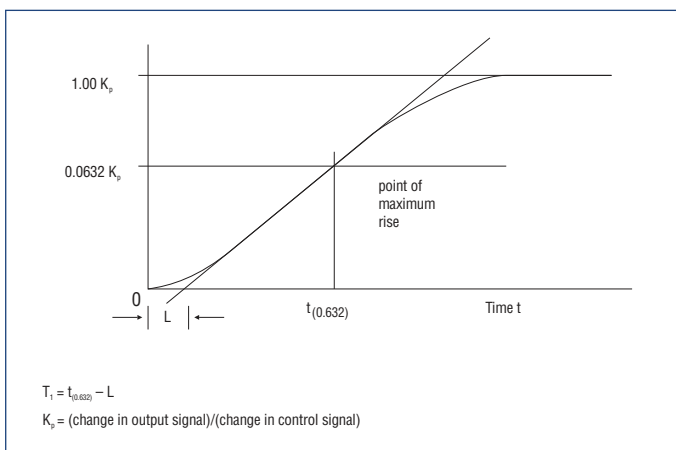


Fig. 6.8 Process response to a step change with $t = 0$ (open loop, control signal amplified)

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:

$$\frac{\text{Output}}{\text{Input}} = \frac{K_p \cdot 10^{-\frac{L}{s}}}{\tau \cdot s + 1} \quad \text{with (6.8)}$$

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:

$$M(s) = K_c \cdot \left(1 + \frac{s}{T_i} + T_d \cdot s \right) \cdot E(s) \quad (6.9)$$

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

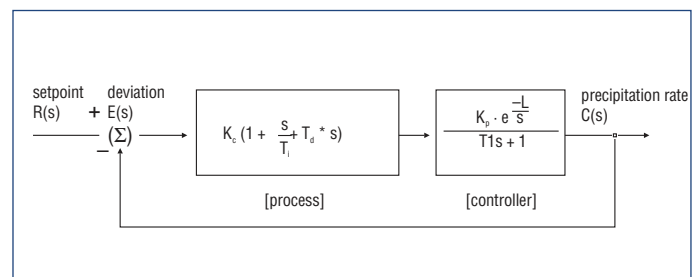


Fig. 6.9 Block diagram of the PID controller

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:

$$ISE = \int e^2(t) \cdot dt \quad (6.10)$$

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:

$$IAE = \int |e(t)| \cdot dt \quad (6.11)$$

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:

$$ITAE = \int t \cdot |e(t)| \cdot dt \quad (6.12)$$

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:

$$K_c = \left(\frac{1.36}{K_p} \right) \cdot \left(\frac{L}{T_1} \right)^{-0.947} \quad (6.13)$$

$$T_i = \left(\frac{1.19}{T_1} \right) \cdot \left(\frac{L}{T_1} \right)^{0.738} \quad (6.14)$$

$$T_d = (0.381 \cdot T_1) \cdot \left(\frac{L}{T_1} \right)^{0.995} \quad (6.15)$$

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

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 nm 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 parameters 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

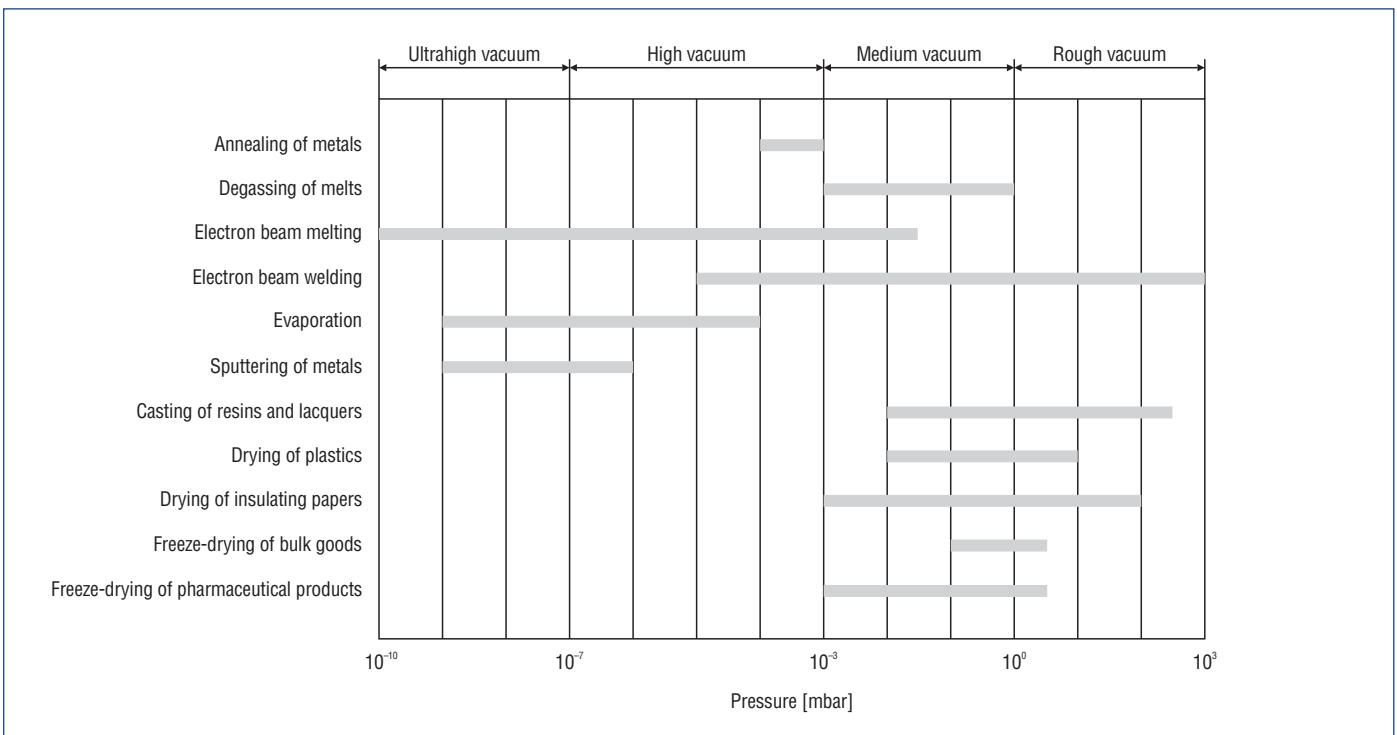


Fig. 7.1 Pressure ranges for various industrial processes

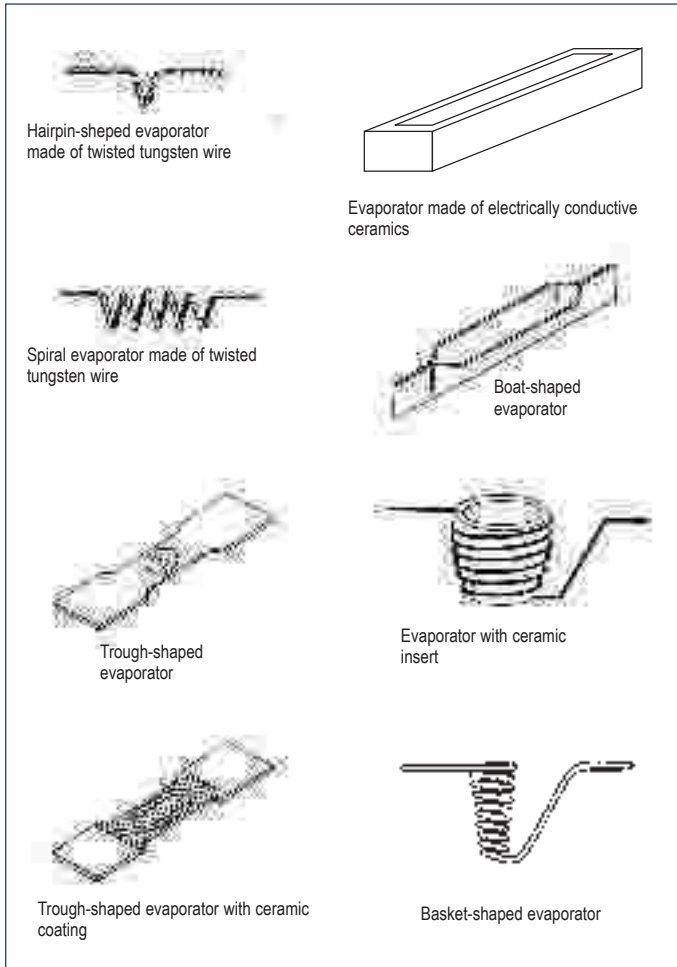


Fig. 7.2 Various thermal evaporators

of material to be heated. In some cases it is not possible to achieve the 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.

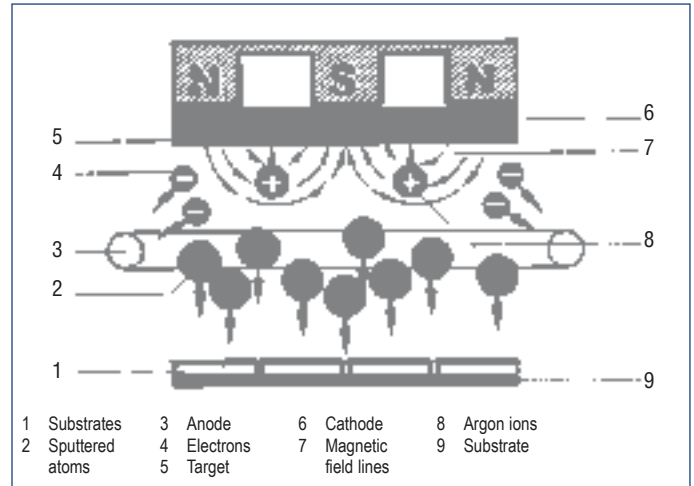


Fig. 7.3 Schematic diagram of a high-performance cathode sputter arrangement

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 activated 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 m² 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.

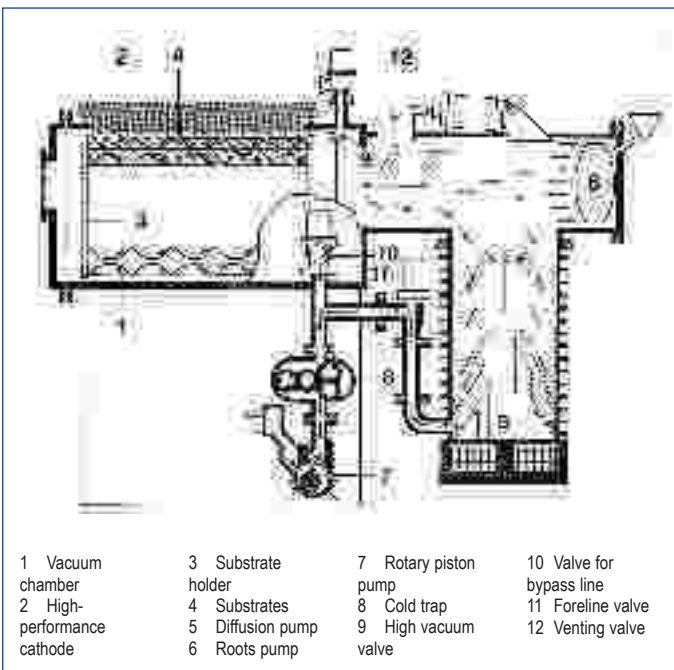


Fig. 7.4 Diagramm of a batch system for coating parts

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 appearance. 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



Fig. 7.5 Multi-chamber parts-coating unit (rotationally symmetric in-line system DynaMet 4V)

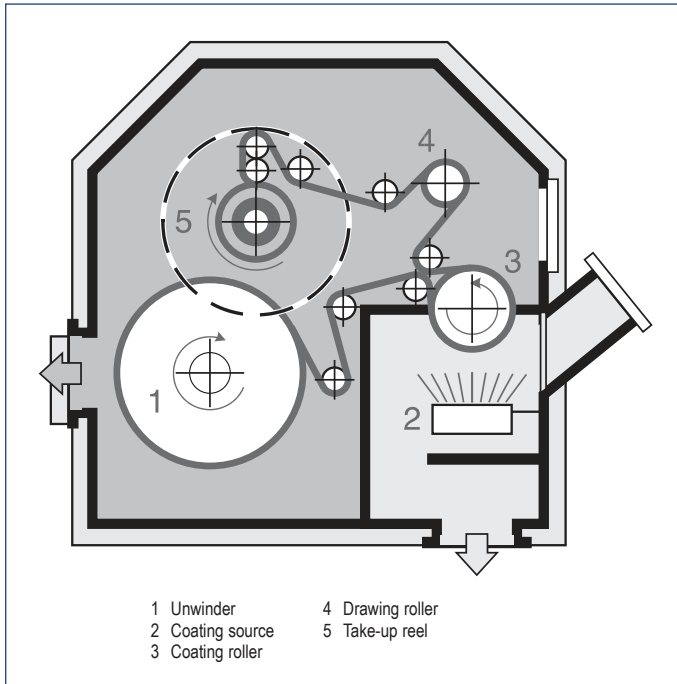


Fig. 7.6 Schematic diagram of a vacuum web coating system

winding system is required for winding and unwinding as well as web guidance.

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

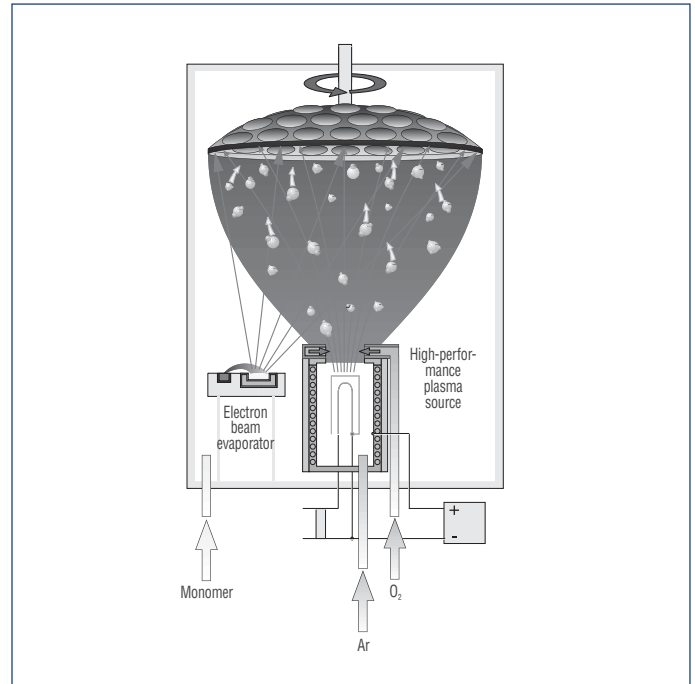


Fig. 7.7 Coating unit for optical coating systems

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.

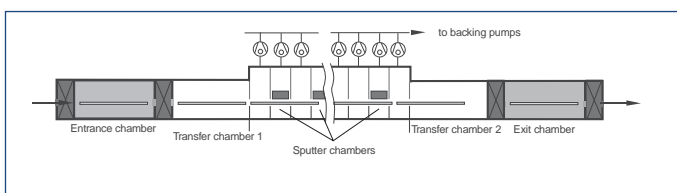


Fig. 7.8 Plant for coating glass panes – 3-chamber in-line system, throughput up to 3,600,000 m² / year

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 separated 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.

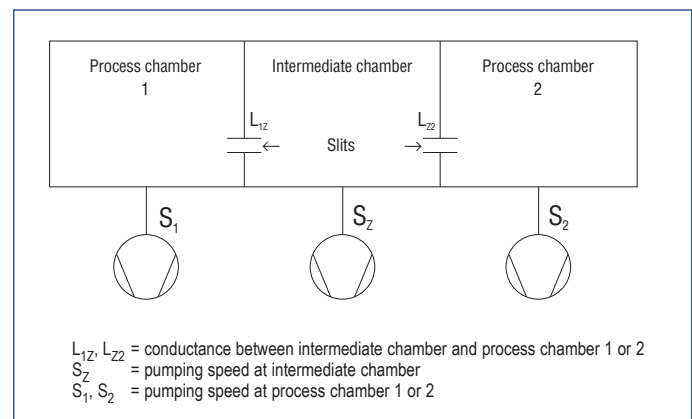


Fig. 7.9 Principle of chamber separation through pressure stages

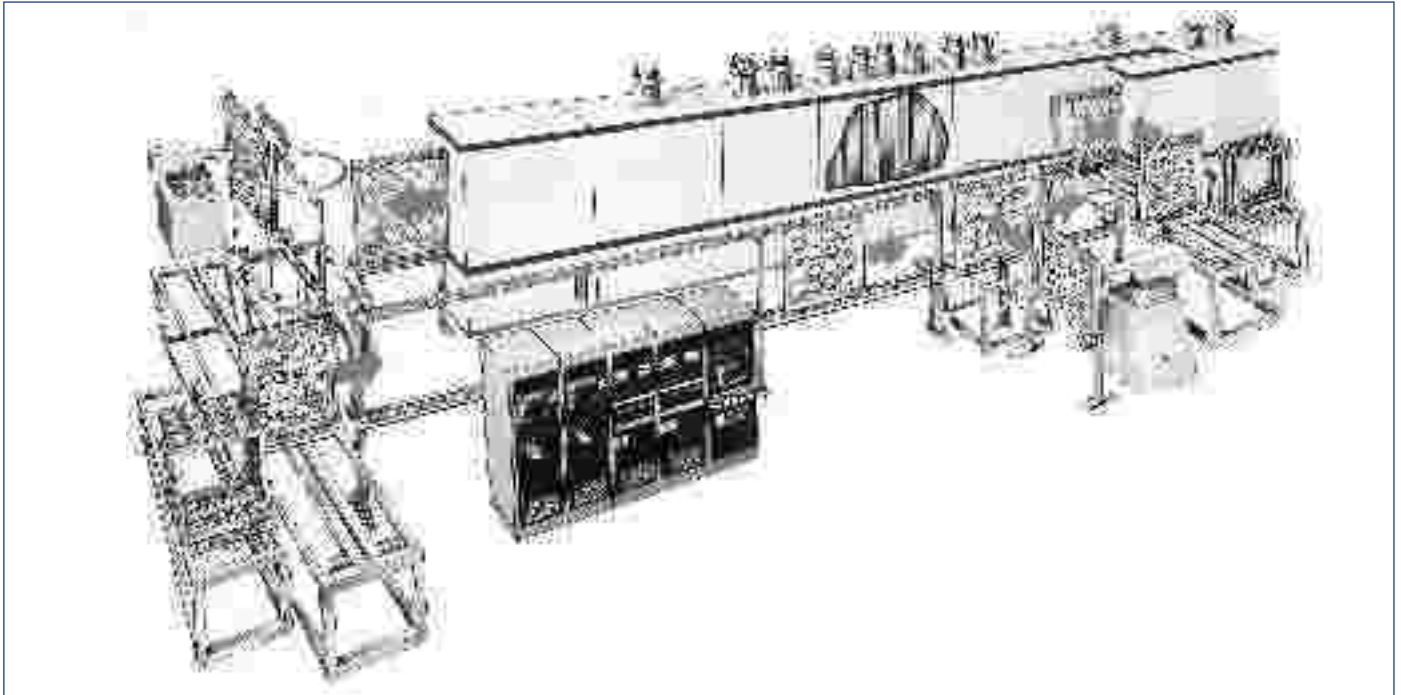


Fig. 7.10 Plant for coating data storage disks with carrier transport system

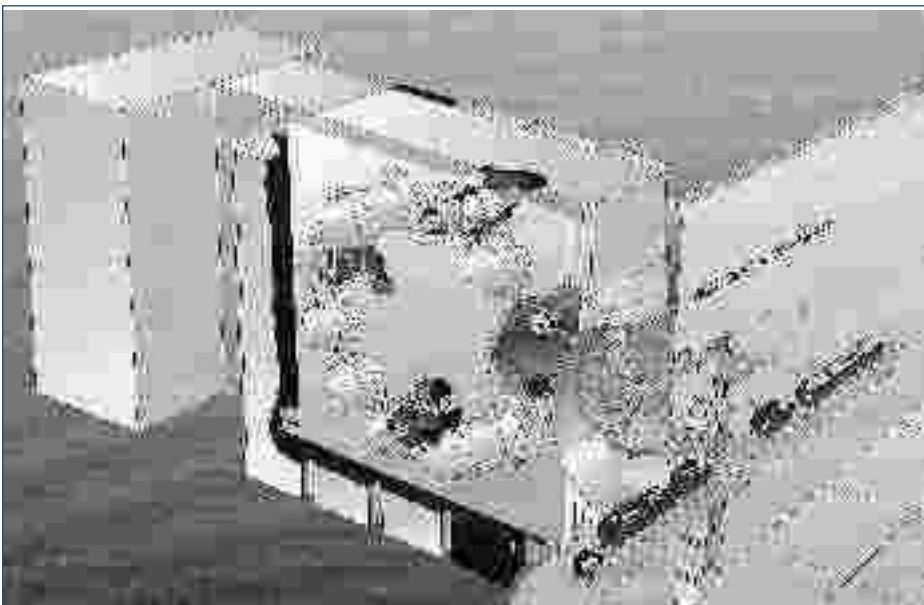


Fig. 7.11 Plant for individual coating of data storage disks

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 be 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.

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.

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 unusable due 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 conditions, 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.

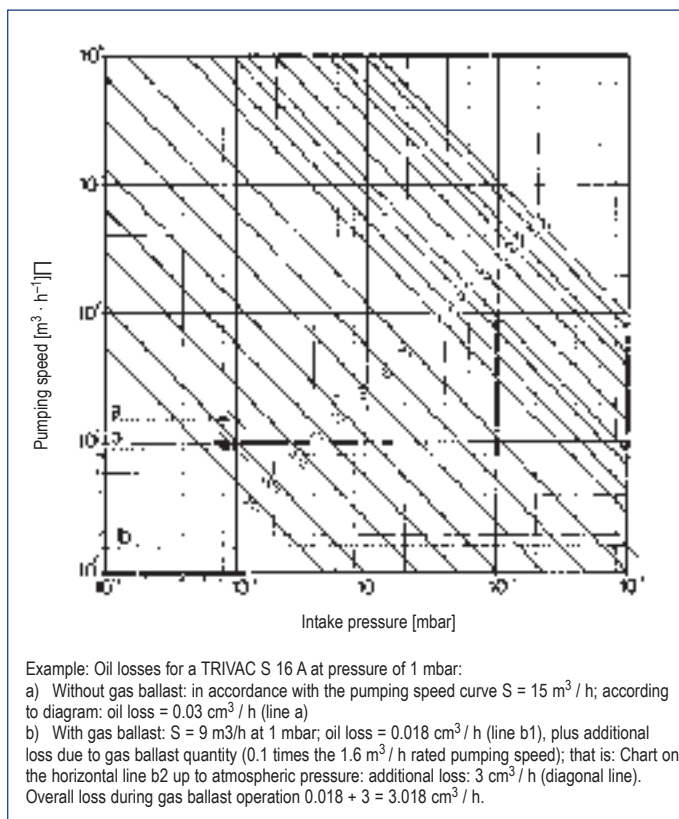


Fig. 8.1 Oil loss for oil-sealed pumps (referenced to an approximate maximum value of 2 cm³ oil loss per cubic meter of air drawn in [STP])

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 N₂). Inert gas ballast should also be used when pumping SO₂, SO₃, and H₂S. A corrosion inhibiting oil is also to be used when handling these three anhydrides. Carbon dioxide (CO₂) 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 maintenance 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 terminal

strip; 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 excessive 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:

$$p \cdot T = 45 \cdot 10^{-3} \text{ mbar} \cdot \text{h}$$

(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 sensor and 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 over-length cables can be obtained from our technical consulting department.

9. Tables, formulas, nomograms and symbols

Unit	$N \cdot m^{-2}, Pa^2)$	mbar	bar	Torr
$1 N \cdot m^{-2} (= 1 Pa)$	1	$1 \cdot 10^{-2}$	$1 \cdot 10^{-5}$	$7.5 \cdot 10^{-3}$
1 mbar	100	1	$1 \cdot 10^{-3}$	0.75
1 bar	$1 \cdot 10^5$	$1 \cdot 10^3$	1	750
1 Torr ³⁾	133	1.33	$1.33 \cdot 10^{-3}$	1

- 1) The torr is included in the table only to facilitate the transition from this familiar unit to the statutory units $N \cdot m^{-2}$, 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 ($N \cdot m^{-2}$) is also designated as Pascal (Pa): $1 N \cdot m^{-2} = 1 Pa$.
 Newton divided by square meters or Pascal is the SI unit for the pressure of fluids.
 3) 1 torr = 4/3 mbar; 1 torr = 1 mbar.

Abbrev.	Gas	$C^* = \lambda \cdot p$ [cm · mbar]
H ₂	Hydrogen	$12.00 \cdot 10^{-3}$
He	Helium	$18.00 \cdot 10^{-3}$
Ne	Neon	$12.30 \cdot 10^{-3}$
Ar	Argon	$6.40 \cdot 10^{-3}$
Kr	Krypton	$4.80 \cdot 10^{-3}$
Xe	Xenon	$3.60 \cdot 10^{-3}$
Hg	Mercury	$3.05 \cdot 10^{-3}$
O ₂	Oxygen	$6.50 \cdot 10^{-3}$
N ₂	Nitrogen	$6.10 \cdot 10^{-3}$
HCl	Hydrochloric acid	$4.35 \cdot 10^{-3}$
CO ₂	Carbon dioxide	$3.95 \cdot 10^{-3}$
H ₂ O	Water vapor	$3.95 \cdot 10^{-3}$
NH ₃	Ammonia	$4.60 \cdot 10^{-3}$
C ₂ H ₅ OH	Ethanol	$2.10 \cdot 10^{-3}$
Cl ₂	Chlorine	$3.05 \cdot 10^{-3}$
Air	Air	$6.67 \cdot 10^{-3}$

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 ²)	dyn · cm ⁻² (μbar)	atm (phys.)	Torr (mm Hg)	inch Hg	Micron (μ)	cm H ₂ O	kp · cm ⁻² (at tech.)	lb · in ⁻² (psi)	lb · ft ⁻²
mbar	1	10 ²	10 ³	$9.87 \cdot 10^{-4}$	0.75	$2.953 \cdot 10^{-2}$	$7.5 \cdot 10^2$	1.02	$1.02 \cdot 10^{-3}$	$1.45 \cdot 10^{-2}$	2.089
Pa	10 ⁻²	1	10	$9.87 \cdot 10^{-6}$	$7.5 \cdot 10^{-3}$	$2.953 \cdot 10^{-4}$	7.5	$1.02 \cdot 10^{-2}$	$1.02 \cdot 10^{-5}$	$1.45 \cdot 10^{-4}$	$2.089 \cdot 10^{-2}$
μbar	10 ⁻³	0.1	1	$9.87 \cdot 10^{-7}$	$7.5 \cdot 10^{-4}$	$2.953 \cdot 10^{-5}$	$7.5 \cdot 10^{-1}$	$1.02 \cdot 10^{-3}$	$1.02 \cdot 10^{-6}$	$1.45 \cdot 10^{-5}$	$2.089 \cdot 10^{-3}$
atm	1013	$1.01 \cdot 10^5$	$1.01 \cdot 10^6$	1	760	29.92	$7.6 \cdot 10^5$	$1.03 \cdot 10^3$	1.033	14.697	2116.4
Torr	1.33	$1.33 \cdot 10^2$	$1.33 \cdot 10^3$	$1.316 \cdot 10^{-3}$	1	$3.937 \cdot 10^{-2}$	10 ³	1.3595	$1.36 \cdot 10^{-3}$	$1.934 \cdot 10^{-2}$	2.7847
in Hg	33.86	$33.9 \cdot 10^2$	$33.9 \cdot 10^3$	$3.342 \cdot 10^{-2}$	25.4	1	$2.54 \cdot 10^4$	34.53	$3.453 \cdot 10^{-2}$	0.48115	70.731
μ	$1.33 \cdot 10^{-3}$	$1.33 \cdot 10^{-1}$	1.333	$1.316 \cdot 10^{-6}$	10 ⁻³	$3.937 \cdot 10^{-5}$	1	$1.36 \cdot 10^{-3}$	$1.36 \cdot 10^{-6}$	$1.934 \cdot 10^{-5}$	$2.785 \cdot 10^{-3}$
cm H ₂ O	0.9807	98.07	980.7	$9.678 \cdot 10^{-4}$	0.7356	$2.896 \cdot 10^{-2}$	$7.36 \cdot 10^2$	1	10 ⁻³	$1.422 \cdot 10^{-2}$	2.0483
at	$9.81 \cdot 10^2$	$9.81 \cdot 10^4$	$9.81 \cdot 10^5$	0.968	$7.36 \cdot 10^2$	28.96	$7.36 \cdot 10^5$	103	1	14.22	2048.3
psi	68.95	$68.95 \cdot 10^2$	$68.95 \cdot 10^3$	$6.804 \cdot 10^{-2}$	51.71	2.036	$51.71 \cdot 10^3$	70.31	$7.03 \cdot 10^{-2}$	1	$1.44 \cdot 10^2$
lb · ft ⁻²	0.4788	47.88	478.8	$4.725 \cdot 10^{-4}$	0.3591	$1.414 \cdot 10^{-2}$	359.1	0.488	$4.88 \cdot 10^{-4}$	$6.94 \cdot 10^{-3}$	1

Normal conditions: 0 °C and sea level, i.e. $p = 1013 \text{ mbar} = 760 \text{ mm Hg} = 760 \text{ torr} = 1 \text{ atm}$
 in Hg = inches of mercury; 1 mtorr (millitorr) = $10^{-3} \text{ torr} = 1 \mu$ (micron ... μm Hg column)
 Pounds per square inch = $lb \cdot in^{-2} = lb / sqin = psi$ (psig = psi gauge ... pressure above atmospheric, pressure gauge reading; psia = psi absolute ... absolute pressure)
 Pounds per square foot = $lb / sqft = lb / ft^2$; $kgf/sqcm^2 = kg \text{ force per square cm} = kp / cm^2 = at$; analogously also: $lbf / sqin = psi$
 1 dyn · cm⁻² (cgs) = 1 μbar (microbar) = 1 barye; 1 bar = 0.1 Mpa; 1 cm water column (cm water column = g / cm² at 4 °C) = 1 Ger (Geryk)
 atm ... physical atmosphere – at ... technical atmosphere; $100 - (x \text{ mbar} / 10.13) = y \% \text{ vacuum}$

Table II: Conversion of pressure units

Variable	General formula	For easy calculation	Value for air at 20°C
Most probable speed of particles c_w	$c_w = \sqrt{\frac{2 \cdot R \cdot T}{M}}$	$c_w = 1.29 \cdot 10^4 \sqrt{\frac{T}{M}} \left[\frac{\text{cm}}{\text{s}} \right]$	$c_w = 410 \text{ [m/s]}$
Mean velocity of particles \bar{c}	$\bar{c} = \sqrt{\frac{8 \cdot R \cdot T}{\pi \cdot M}}$	$\bar{c} = 1.46 \cdot 10^4 \sqrt{\frac{T}{M}} \left[\frac{\text{cm}}{\text{s}} \right]$	$\bar{c} = 464 \text{ [m/s]}$
Mean square of velocity of particles \bar{c}^2	$\bar{c}^2 = \frac{3 \cdot R \cdot T}{M}$	$\bar{c}^2 = 2.49 \cdot 10^8 \frac{T}{M} \left[\frac{\text{cm}^2}{\text{s}^2} \right]$	$\bar{c}^2 = 25.16 \cdot 10^4 \left[\frac{\text{cm}^2}{\text{s}^2} \right]$
Gas pressure p of particles	$p = n \cdot k \cdot T$ $p = \frac{1}{3} \cdot n \cdot m_T \cdot \bar{c}^2$ $p = \frac{1}{3} \cdot \rho \cdot \bar{c}^2$	$p = 13.80 \cdot 10^{-20} \cdot n \cdot T \text{ [mbar]}$	$p = 4.04 \cdot 10^{-17} \cdot n \text{ [mbar]}$ (applies to all gases)
Number density of particles n	$n = p/kT$	$n = 7.25 \cdot 10^{18} \frac{p}{T} \text{ [cm}^{-3}\text{]}$	$p = 2.5 \cdot 10^{16} \cdot p \text{ [cm}^{-3}\text{]}$ (applies to all gases)
Area-related impingement Z_A	$Z_A = \frac{1}{4} \cdot n \cdot \bar{c}$ $Z_A = \sqrt{\frac{N_A}{2 \cdot \pi \cdot M \cdot k \cdot T}} p$	$Z_A = 2.63 \cdot 10^{22} \frac{p}{\sqrt{M \cdot T}} \text{ [cm}^{-2} \text{ s}^{-1}\text{]}$	$Z_A = 2.85 \cdot 10^{20} \cdot p \text{ [cm}^{-2} \text{ s}^{-1}\text{]}$ (see Fig. 78.2)
Volume collision rate Z_V	$Z_V = \frac{1}{2} \frac{n \cdot \bar{c}}{\lambda}$ $Z_A = \frac{1}{c^*} \sqrt{\frac{2 \cdot N_A}{\pi \cdot M \cdot k \cdot T}} p^2$	$Z_V = 5.27 \cdot 10^{22} \frac{p^2}{c^* \cdot \sqrt{M \cdot T}} \text{ [cm}^{-3} \text{ s}^{-1}\text{]}$	$Z_V = 8.6 \cdot 10^{22} \cdot p^2 \text{ [cm}^{-3} \text{ s}^{-1}\text{]}$ (see Fig. 78.2)
Equation of state of ideal gas	$p \cdot V = v \cdot R \cdot T$	$p \cdot V = 83.14 \cdot v \cdot T \text{ [mbar} \cdot \ell\text{]}$	$p \cdot V = 2.44 \cdot 10^4 v \text{ [mbar} \cdot \ell\text{]}$ (for all gases)
Area-related mass flow rate $q_{m,A}$	$q_{m,A} = Z_A \cdot m_T = \sqrt{\frac{M}{2 \cdot \pi \cdot k \cdot T}} \cdot N_A \cdot p$	$Q_{m,A} = 4.377 \cdot 10^{-2} \sqrt{\frac{M}{T}} \cdot p \text{ [g cm}^{-2} \text{ s}^{-1}\text{]}$	$q_{m,A} = 1.38 \cdot 10^{-2} \cdot p \text{ g [cm}^{-2} \text{ s}^{-1}\text{]}$

$c^* = \lambda \cdot p$ in $\text{cm} \cdot \text{mbar}$ (see Tab. III)
 k Boltzmann constant in $\text{mbar} \cdot \ell \cdot \text{K}^{-1}$
 λ mean free path in cm
 M molar mass in $\text{g} \cdot \text{mol}^{-1}$
 m_T particle mass in g
 N_A Avogadro constant in mol^{-1}
 n number density of particles in cm^{-3}
 v amount of substance in mol
 p gas pressure in mbar
 R molar gas constant in $\text{mbar} \cdot \ell \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$
 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	$1.6605 \cdot 10^{-27} \text{ kg}$	
Avogadro constant	N_A	$6.0225 \cdot 10^{23} \text{ mol}^{-1}$	Number of particles per mol, formerly: Loschmidt number
Boltzmann constant	k	$1.3805 \cdot 10^{-23} \text{ J} \cdot \text{K}^{-1}$ $13.805 \cdot 10^{-23} \frac{\text{mbar} \cdot \ell}{\text{K}}$	
Electron rest mass	m_e	$9.1091 \cdot 10^{-31} \text{ kg}$	
Elementary charge	e	$1.6021 \cdot 10^{-19} \text{ A} \cdot \text{s}$	
Molar gas constant	R	$8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ $= 83.14 \frac{\text{mbar} \cdot \ell}{\text{mol} \cdot \text{K}}$	$R = N_A \cdot k$
Molar volume of the ideal gas	V_o	$22.414 \text{ m}^3 \text{ kmol}^{-1}$ $22.414 \text{ l} \cdot \text{mol}^{-1}$	DIN 1343; formerly: molar volume at 0 °C and 1013 mbar
Standard acceleration of free fall	g_n	$9.8066 \text{ m} \cdot \text{s}^{-2}$	
Planck constant	h	$6.6256 \cdot 10^{-34} \text{ J} \cdot \text{s}$	
Stefan-Boltzmann constant	σ	$5.669 \cdot 10^{-8} \frac{\text{W}}{\text{m}^2 \text{ K}^4}$	also: unit conductance, radiation constant
Specific electron charge	$\frac{-e}{m_e}$	$-1.7588 \cdot 10^{11} \frac{\text{A} \cdot \text{s}}{\text{kg}}$	
Speed of light in vacuum	c	$2.9979 \cdot 10^8 \text{ m} \cdot \text{s}^{-1}$	
Standard reference density of a gas	ρ_n	$\text{kg} \cdot \text{m}^{-3}$	Density at $\vartheta = 0 \text{ °C}$ and $p_n = 1013 \text{ mbar}$
Standard reference pressure	p_n	$101.325 \text{ Pa} = 1013 \text{ mbar}$	DIN 1343 (Nov. 75)
Standard reference temperature	T_n	$T_n = 273.15 \text{ K}, \vartheta = 0 \text{ °C}$	DIN 1343 (Nov. 75)

Table V: Important values

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

$1 \downarrow = \dots \rightarrow$	mbar · l/s	kg · h ⁻¹ (20 °C)	kg · h ⁻¹ (0 °C)	cm ³ /h (NTP)	cm ³ /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	$1.56 \cdot 10^5$	$1.54 \cdot 10^5$	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
cm ³ /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	–	$44.7 \cdot 10^{-2}$	$2.11 \cdot 10^{-1}$	$2.81 \cdot 10^{-2}$	$1.66 \cdot 10^{-5}$
cm ³ /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

$1 cm^3 (NTP) = 1 cm^3$ under normal conditions ($T = 273.15 K$; $p = 1013.25 mbar$)
 $1 cm^3 (NTP) \cdot h^{-1} = 1 atm \cdot cm^3 \cdot h^{-1} = 1 Ncm^3 \cdot h^{-1} = 1 std cch$
 $1 cm^3 (NTP) \cdot s^{-1} = 1 sccs = 60 cm^3 (NTP) \cdot min^{-1} = 60 sccm = 60 std ccm = 60 Ncm^3 \cdot min^{-1}$
 Freon F 12 (CCl_2F_2) $M = 120.92 g \cdot mol^{-1}$; air $M = 28.96 g \cdot mol^{-1}$
 Note: Anglo-American units are not abbreviated nonuniformly! Example: Standard cubic centimeter per minute \rightarrow sccm = sccpm = std ccm = std ccpm
 NTP = normal temperature and pressure (1 atm; 0 °C) $R = 83.14 mbar \cdot l \cdot mol^{-1} \cdot K^{-1}$
 SI coherent: $1 Pa \cdot m^3 \cdot s^{-1} = 10 mbar \cdot l \cdot s^{-1}$; $R = 8.314 Pa \cdot m^3 \cdot mol^{-1} \cdot K^{-1}$; M in kg / mol
 $1 lusec = 1 l \cdot \mu \cdot s^{-1}$ $1 \cdot \mu = 1 micron = 10^{-3} Torr$ $1 lusec = 10^{-3} Torr \cdot l \cdot s^{-1}$
 $1 sccm = 10^{-3} slpm = 10^{-3} N \cdot l \cdot min^{-1} = 60 sccs$

Table VIIa: Conversion of throughput (Q_{pV}) units; (leak rate) units

$1 \downarrow = \dots \rightarrow$	mbar · l/s	cm ³ /s **)	Torr · l/s	Pa · m ³ /s	g/a *)	oz/yr *)	lb/yr *)	atm · ft ³ /min	μ · l/s	μ · ft ³ /h	μ · ft ³ /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
cm ³ /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 · m ³ /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	$6.18 \cdot 10^{-2}$	$3.80 \cdot 10^{-7}$	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 · ft ³ /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 · ft ³ /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 · ft ³ /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

$1 \cdot \mu \cdot ft^3 \cdot h^{-1} = 1.04 \cdot 10^{-5} std cc per second$
 $1 cm^3 \cdot s^{-1} (NTP) = 1 atm \cdot cm^3 \cdot s^{-1} = 1 scc \cdot s^{-1} = 1 sccss$
 $1 atm \cdot ft^3 \cdot min^{-1} = 1 cfm (NTP)$
 $1 Pa \cdot m^3/s = 1 Pa \cdot m^3/s (anglo-am.) = 10^3 Pa \cdot l/s$
 *) F12 (20 °C) $C.Cl_2F_2$ $M = 120.92 h/mol$
 **) (NTP) normal temperature and pressure 1 atm und 0 °C
 $1 micron cubic foot per hour = 0.0079 micron liter per second$
 $1 micron liter per second = 0.0013 std cc per second = 1 lusec$
 $1 micron cubic foot per minute = 1 \mu \cdot ft^3 \cdot min^{-1} = 1 \mu \cdot cuft \cdot min^{-1} = 1 \mu \cdot cfm$
 $1 standard cc per second = 96.600 micron cubic feet per hour$
 $1 \mu \cdot l \cdot s^{-1} = 127 \mu \cdot ft^3 \cdot h^{-1} = 0.0013 std cc per second = 1 lusec$
 $1 kg = 2.2046 pounds (lb)$
 $1 cubic foot (cfut. cf) = 28.3168 dm^3$
 $1 lb = 16 ounces (oz)$
 $1 lusec = 1 \mu \cdot l \cdot s^{-1}$
 $1 std cc/sec = 760 \mu \cdot l \cdot s^{-1}$

Table VII b: Conversion of throughput (Q_{pV}) units; (leak rate) units

	% 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 · 10 ⁻³	1.8 · 10 ⁻³	1.9 · 10 ⁻²
He	7 · 10 ⁻⁵	7 · 10 ⁻⁵	5.3 · 10 ⁻³
CH ₄	2 · 10 ⁻⁴	2 · 10 ⁻⁴	2 · 10 ⁻³
Kr	3 · 10 ⁻⁴	1.1 · 10 ⁻⁴	1.1 · 10 ⁻³
N ₂ O	6 · 10 ⁻⁵	5 · 10 ⁻⁵	5 · 10 ⁻⁴
H ₂	5 · 10 ⁻⁶	5 · 10 ⁻⁵	5 · 10 ⁻⁴
Xe	4 · 10 ⁻⁵	8.7 · 10 ⁻⁶	9 · 10 ⁻⁵
O ₃	9 · 10 ⁻⁶	7 · 10 ⁻⁶	7 · 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 · s ⁻¹]	10 ²³ – 10 ²⁰	10 ²⁰ – 10 ¹⁷	10 ¹⁷ – 10 ¹³	< 10 ¹³
Vol.-related collision rate	Z _v [cm ⁻³ · 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 extent 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 · l · s ⁻¹ · cm ⁻²)		Metals 10 ⁻⁹ ... · 10 ⁻⁷				Nonmetals 10 ⁻⁷ ... · 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 · 10 ⁻⁸	1.1 · 10 ⁻⁸	2 · 10 ⁻⁹		Silicone	1.5 · 10 ⁻⁵	8 · 10 ⁻⁶	3.5 · 10 ⁻⁶	1.5 · 10 ⁻⁶
Al	2 · 10 ⁻⁸	6 · 10 ⁻⁹			NBR	4 · 10 ⁻⁶	3 · 10 ⁻⁶	1.5 · 10 ⁻⁶	1 · 10 ⁻⁶
Cu	4 · 10 ⁻⁸	2 · 10 ⁻⁸	6 · 10 ⁻⁹	3.5 · 10 ⁻⁹	Acrylic glass	1.5 · 10 ⁻⁶	1.2 · 10 ⁻⁶	8 · 10 ⁻⁷	5 · 10 ⁻⁷
Stainless steel		9 · 10 ⁻⁸	3.5 · 10 ⁻⁸	2.5 · 10 ⁻⁸	FPM, FKM	7 · 10 ⁻⁷	4 · 10 ⁻⁷	2 · 10 ⁻⁷	1.5 · 10 ⁻⁷

¹ All values depend largely on pretreatment!

Table X: Outgassing rate of materials in mbar · l · s⁻¹ · cm⁻²

Nominal width (DN)	Internal diam. (mm)	
Series	R5	R10
	10	10
	16	16
		20
	25	24
		32
	40	41
		50
	63	70
		80
	100	102
		125
	160	153
		200
	250	261
		320
	400	400
		500
	630	651
		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 PNEUROPE).

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

t °C	ps mbar	ρ_D g/m ³	t °C	ps mbar	ρ_D g/m ³	t °C	ps mbar	ρ_D g/m ³	t °C	ps mbar	ρ_D g/m ³
-100	1.403 · 10 ⁻⁵	1.756 · 10 ⁻⁵	-35	0.2233	0.2032	30	42.43	30.38	95	845.3	504.5
-99	1.719	2.139	-34	0.2488	0.2254	31	44.93	32.07	96	876.9	522.1
-98	2.101	2.599	-33	0.2769	0.2498	32	47.55	33.83	97	909.4	540.3
-97	2.561	3.150	-32	0.3079	0.2767	33	50.31	35.68	98	943.0	558.9
-96	3.117	3.812	-31	0.3421	0.3061	34	53.20	37.61	99	977.6	578.1
-95	3.784 · 10 ⁻⁵	4.602 · 10 ⁻⁵	-30	0.3798	0.3385	35	56.24	39.63	100	1013.2	597.8
-94	4.584	5.544	-29	0.4213	0.3739	36	59.42	41.75	101	1050	618.0
-93	5.542	6.665	-28	0.4669	0.4127	37	62.76	43.96	102	1088	638.8
-92	6.685	7.996	-27	0.5170	0.4551	38	66.26	46.26	103	1127	660.2
-91	8.049	9.574	-26	0.5720	0.5015	39	69.93	48.67	104	1167	682.2
-90	9.672 · 10 ⁻⁵	11.44 · 10 ⁻⁵	-25	0.6323	0.5521	40	73.78	51.19	105	1208	704.7
-89	11.60	13.65	-24	0.6985	0.6075	41	77.80	53.82	106	1250	727.8
-88	13.88	16.24	-23	0.7709	0.6678	42	82.02	56.56	107	1294	751.6
-87	16.58	19.30	-22	0.8502	0.7336	43	86.42	59.41	108	1339	776.0
-86	19.77	22.89	-21	0.9370	0.8053	44	91.03	62.39	109	1385	801.0
-85	23.53 · 10 ⁻⁵	27.10 · 10 ⁻⁵	-20	1.032	0.8835	45	95.86	65.50	110	1433	826.7
-84	27.96	32.03	-19	1.135	0.9678	46	100.9	68.73	111	1481	853.0
-83	33.16	37.78	-18	1.248	1.060	47	106.2	72.10	112	1532	880.0
-82	39.25	44.49	-17	1.371	1.160	48	111.7	75.61	113	1583	907.7
-81	46.38	52.30	-16	1.506	1.269	49	117.4	79.26	114	1636	936.1
-80	0.5473 · 10 ⁻³	0.6138 · 10 ⁻³	-15	1.652	1.387	50	123.4	83.06	115	1691	965.2
-79	0.6444	0.7191	-14	1.811	1.515	51	129.7	87.01	116	1746	995.0
-78	0.7577	0.8413	-13	1.984	1.653	52	136.2	91.12	117	1804	1026
-77	0.8894	0.9824	-12	2.172	1.803	53	143.0	95.39	118	1863	1057
-76	1.042	1.145	-11	2.376	1.964	54	150.1	99.83	119	1923	1089
-75	1.220 · 10 ⁻³	1.334 · 10 ⁻³	-10	2.597	2.139	55	157.5	104.4	120	1985	1122
-74	1.425	1.550	-9	2.837	2.328	56	165.2	109.2	121	2049	1156
-73	1.662	1.799	-8	3.097	2.532	57	173.2	114.2	122	2114	1190
-72	1.936	2.085	-7	3.379	2.752	58	181.5	119.4	123	2182	1225
-71	2.252	2.414	-6	3.685	2.990	59	190.2	124.7	124	2250	1262
-70	2.615 · 10 ⁻³	2.789 · 10 ⁻³	-5	4.015	3.246	60	199.2	130.2	125	2321	1299
-69	3.032	3.218	-4	4.372	3.521	61	208.6	135.9	126	2393	1337
-68	3.511	3.708	-3	4.757	3.817	62	218.4	141.9	127	2467	1375
-67	4.060	4.267	-2	5.173	4.136	63	228.5	148.1	128	2543	1415
-66	4.688	4.903	-1	5.623	4.479	64	239.1	154.5	129	2621	1456
-65	5.406 · 10 ⁻³	5.627 · 10 ⁻³	0	6.108	4.847	65	250.1	161.2	130	2701	1497
-64	6.225	6.449	1	6.566	5.192	66	261.5	168.1	131	2783	1540
-63	7.159	7.381	2	7.055	5.559	67	273.3	175.2	132	2867	1583
-62	8.223	8.438	3	7.575	5.947	68	285.6	182.6	133	2953	1627
-61	9.432	9.633	4	8.129	6.360	69	298.4	190.2	134	3041	1673
-60	10.80 · 10 ⁻³	10.98 · 10 ⁻³	5	8.719	6.797	70	311.6	198.1	135	3131	1719
-59	12.36	12.51	6	9.347	7.260	71	325.3	206.3	136	3223	1767
-58	14.13	14.23	7	10.01	7.750	72	339.6	214.7	137	3317	1815
-57	16.12	16.16	8	10.72	8.270	73	354.3	223.5	138	3414	1865
-56	18.38	18.34	9	11.47	8.819	74	369.6	232.5	139	3512	1915
-55	20.92 · 10 ⁻³	20.78 · 10 ⁻³	10	12.27	9.399	75	385.5	241.8	140	3614	1967
-54	23.80	23.53	11	13.12	10.01	76	401.9	251.5			
-53	27.03	26.60	12	14.02	10.66	77	418.9	261.4			
-52	30.67	30.05	13	14.97	11.35	78	436.5	271.7			
-51	34.76	33.90	14	15.98	12.07	79	454.7	282.3			
-50	39.35 · 10 ⁻³	38.21 · 10 ⁻³	15	17.04	12.83	80	473.6	293.3			
-49	44.49	43.01	16	18.17	13.63	81	493.1	304.6			
-48	50.26	48.37	17	19.37	14.48	82	513.3	316.3			
-47	56.71	54.33	18	20.63	15.37	83	534.2	328.3			
-46	63.93	60.98	19	21.96	16.31	84	555.7	340.7			
-45	71.98 · 10 ⁻³	68.36 · 10 ⁻³	20	23.37	17.30	85	578.0	353.5			
-44	80.97	76.56	21	24.86	18.34	86	601.0	366.6			
-43	90.98	85.65	22	26.43	19.43	87	624.9	380.2			
-42	102.1	95.70	23	28.09	20.58	88	649.5	394.2			
-41	114.5 · 10 ⁻³	106.9 · 10 ⁻³	24	29.83	21.78	89	674.9	408.6			
-40	0.1283	0.1192	25	31.67	23.05	90	701.1	423.5			
-39	0.1436	0.1329	26	33.61	24.38	91	728.2	438.8			
-38	0.1606	0.1480	27	35.65	25.78	92	756.1	454.5			
-37	0.1794	0.1646	28	37.80	27.24	93	784.9	470.7			
-36	0.2002	0.1829	29	40.06	28.78	94	814.6	487.4			

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 $+140^\circ\text{C}$ 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 Minimum Electrical Spark Gap

² Minimum Ignition Current
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)

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	x		
Air, oily	x	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			
Chlorine, 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		
Diethylphthalate	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	-		
Diocetylphthalate	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	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	x	o		
Ethyl acetate	o	o	o	o	o	x	o		
Ethyl acrylate						o	x		
Ethyl alcohol, denatured	-	-				-	x		
Ethyl alcohol, pure	-	-				-	x	x	
Ethyl chloride	x	-	o					x	-
Ethylene bromide	o	o						x	
Ethylene chloride								x	-
Ethylene dichloride	o	o				-	x		
Ethylene glycol	x	x	o	x	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
Furfural	o	o	o	o	o	x			
Gas oil	x	-				x	x	o	
Generator gas	x	-	x					x	
Glycerine	x	x	x	x	x	x	x		
Glycol	x	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
Heptane	x	-	o					x	x
Hexaldehyde	o	o						x	x
Hexane	x	-	o					x	x
Hydraulic fluids									
Hydraulic oils DIN 51524	x	-	-					x	x
Phosphoric ester HFD	o	o	o					x	
Polyglycol water HFC	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	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
Lighting gas	-	-						x	x
Maleic anhydride								x	
Mercury	x	x						x	x
Methane	x	-						x	-
Methane (pit gas)	x	x						x	x
Methylene chloride	o	o						o	x
Methyl acrylate								o	x
Methyl alcohol (methanol)	-	-	x					o	x
Methyl ethyl ketone	o	o						o	x
Methyl isobutyl ketone	o							o	x
Methyl methacrylate	o	o						o	x
Methyl salicylate	o	o						x	-
Naphtalene	o	o						x	o
Natural gas	-	x	-					x	x
Nitrobenzene	o	o						o	x
Nitrous oxide	x							x	x
Oleic acid	x							x	x
Orange oil	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
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
Pentachlorodiphenyl	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	
Trichloroacetic 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 with 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)

Measurement and gauges

	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








	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)

	Kelvin	Celsius	Réaumur	Fahrenheit	Rankine
Boiling point H ₂ O	373	100	80	212	672
Body temperature 37°C	310	37	30	99	559
Room temperature	293	20	16	68	527
Freezing point H ₂ O	273	0	0	32	492
NaCl/H ₂ O 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)

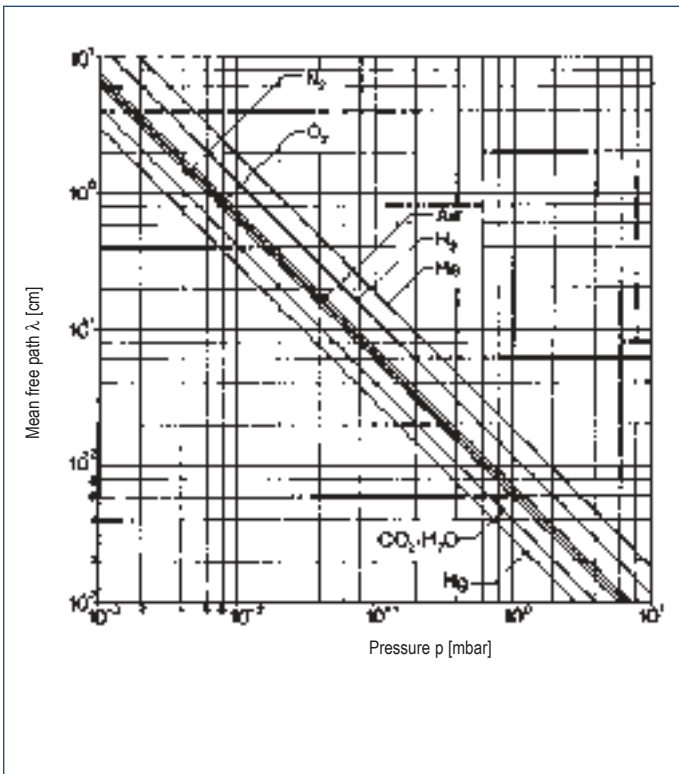


Fig. 9.1: Variation of mean free path λ (cm) with pressure for various gases

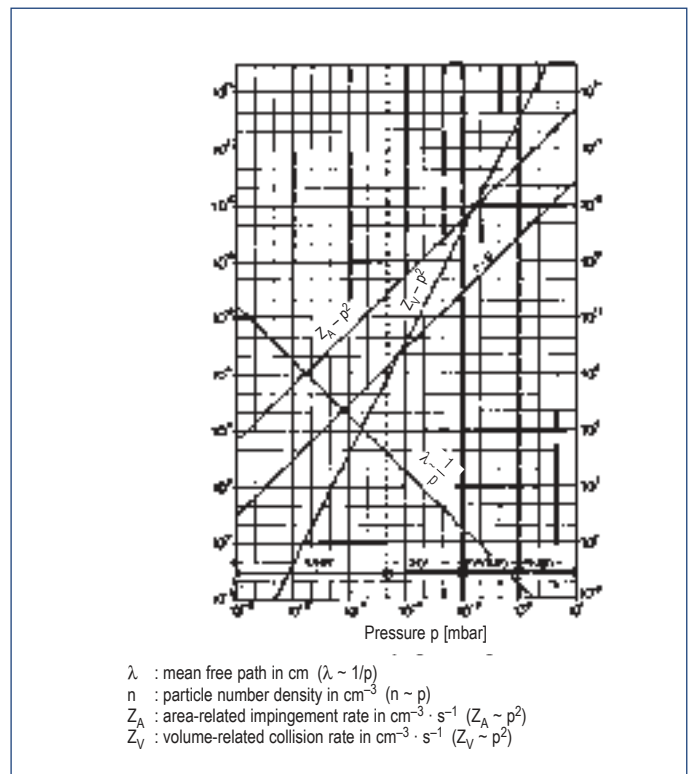


Fig. 9.2: Diagram of kinetics of gases for air at 20 °C

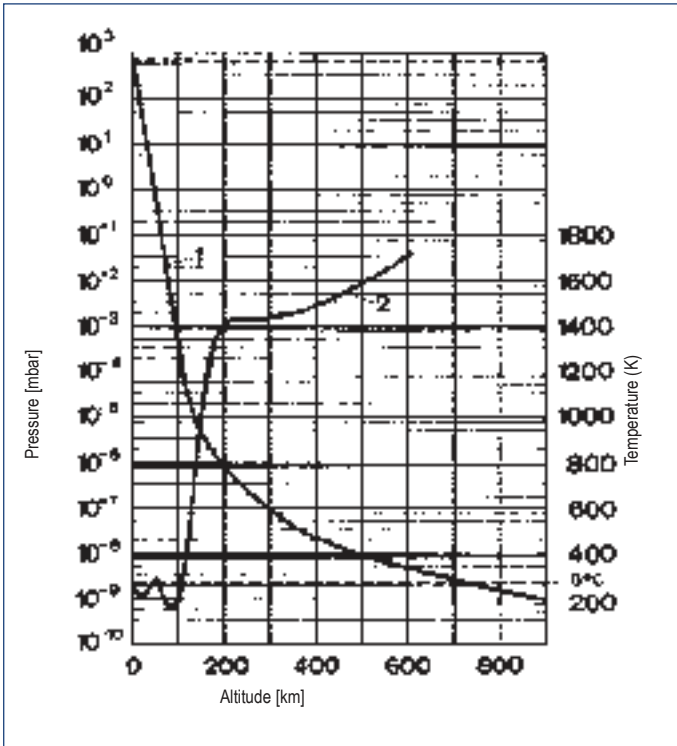


Fig. 9.3: Decrease in air pressure (1) and change in temperature (2) as a function of altitude

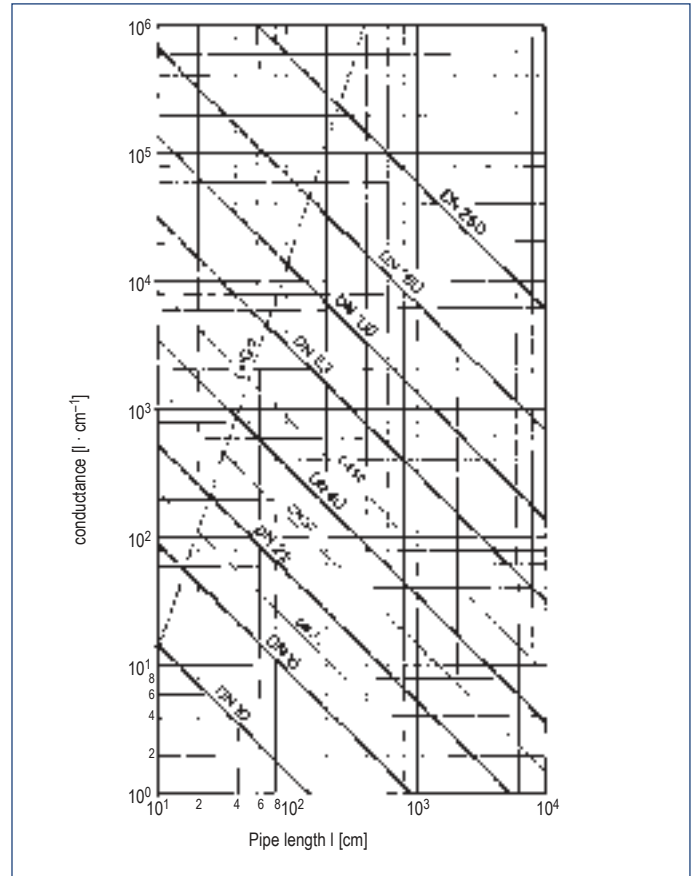


Fig. 9.5: Conductance values for piping of commonly used nominal width with circular cross-section for laminar flow ($p = 1$ mbar) according to equation 53a. (Thick lines refer to preferred DN) Flow medium: air (d, l in cm!)

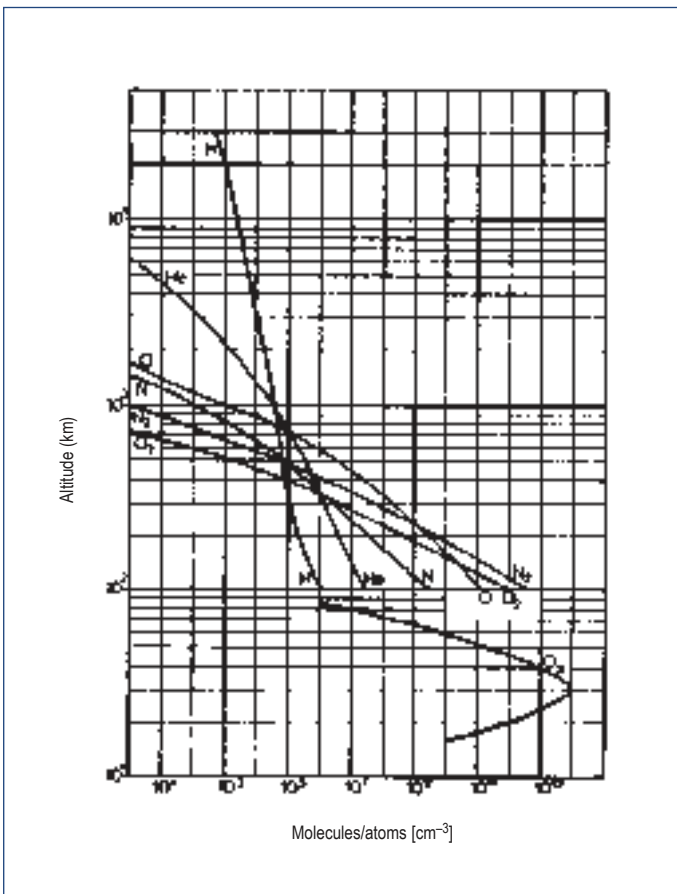


Fig. 9.4: Change in gas composition of the atmosphere as a function of altitude

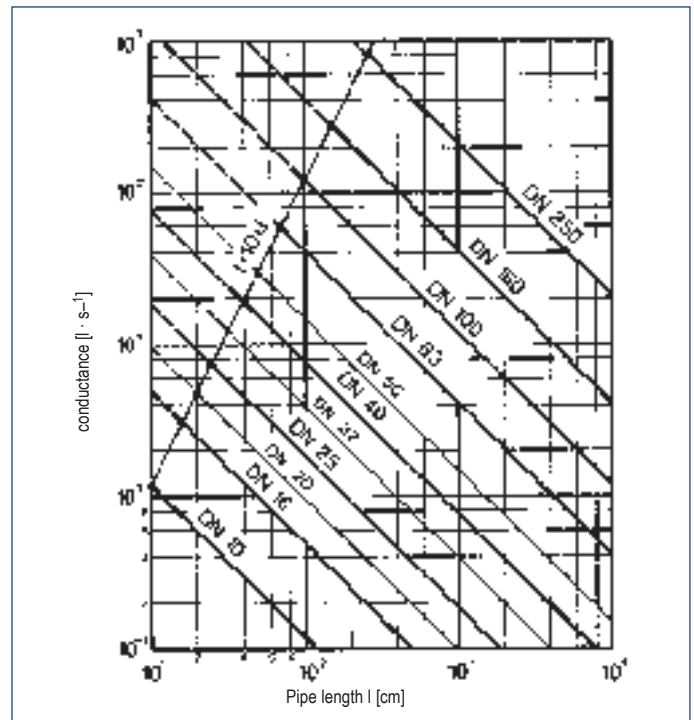
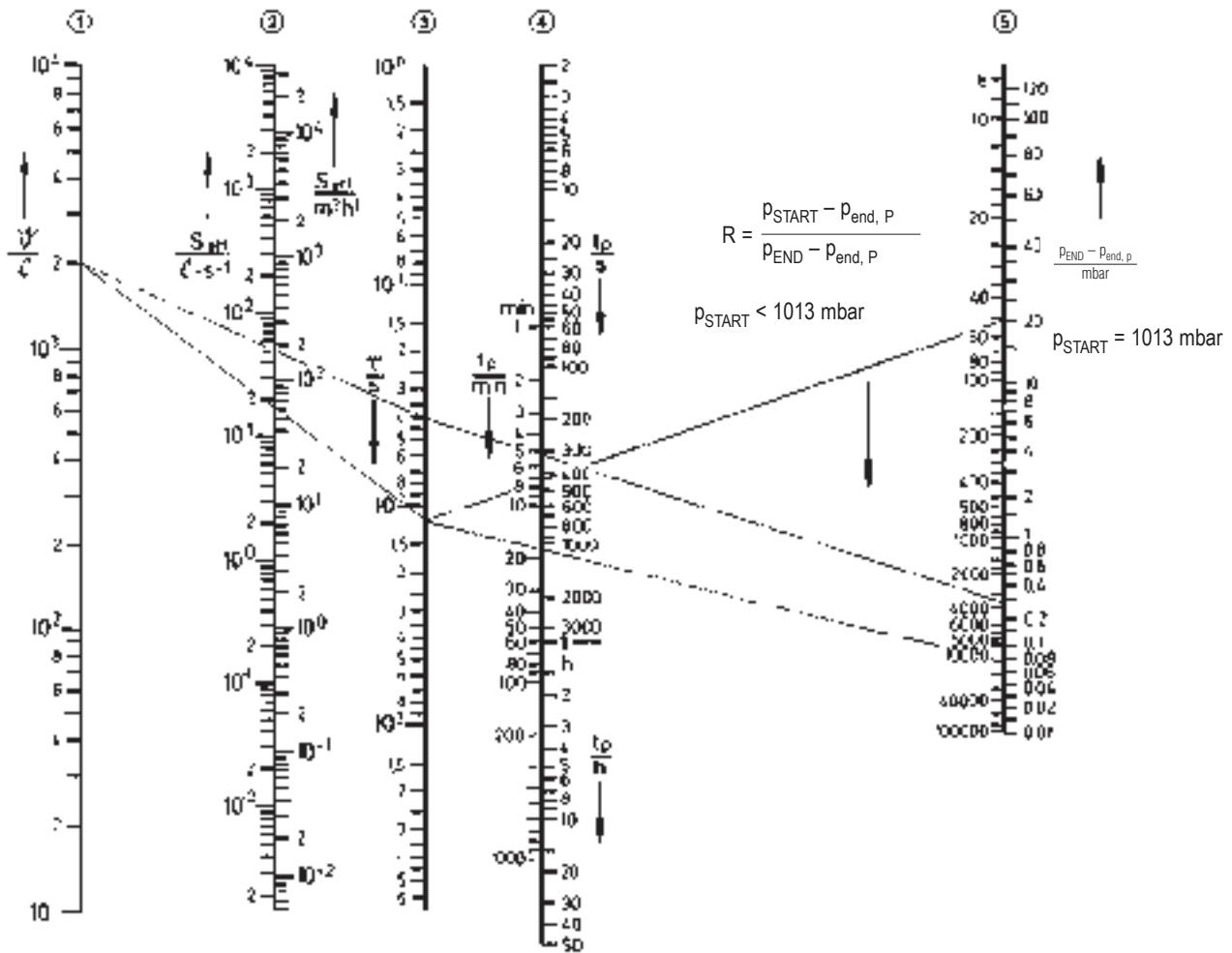


Fig. 9.6: Conductance values for piping of commonly used nominal width with circular cross-section for molecular flow according to equation 53b. (Thick lines refer to preferred DN) Flow medium: air (d, l in cm!)



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} ($p_n = 1013$ mbar) 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 by $p_{ult,p}$. 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 $S_{eff,max} = 60$ m³/h = 16.7 l · s⁻¹. The pump-down time can be obtained from the nomogram in two steps:

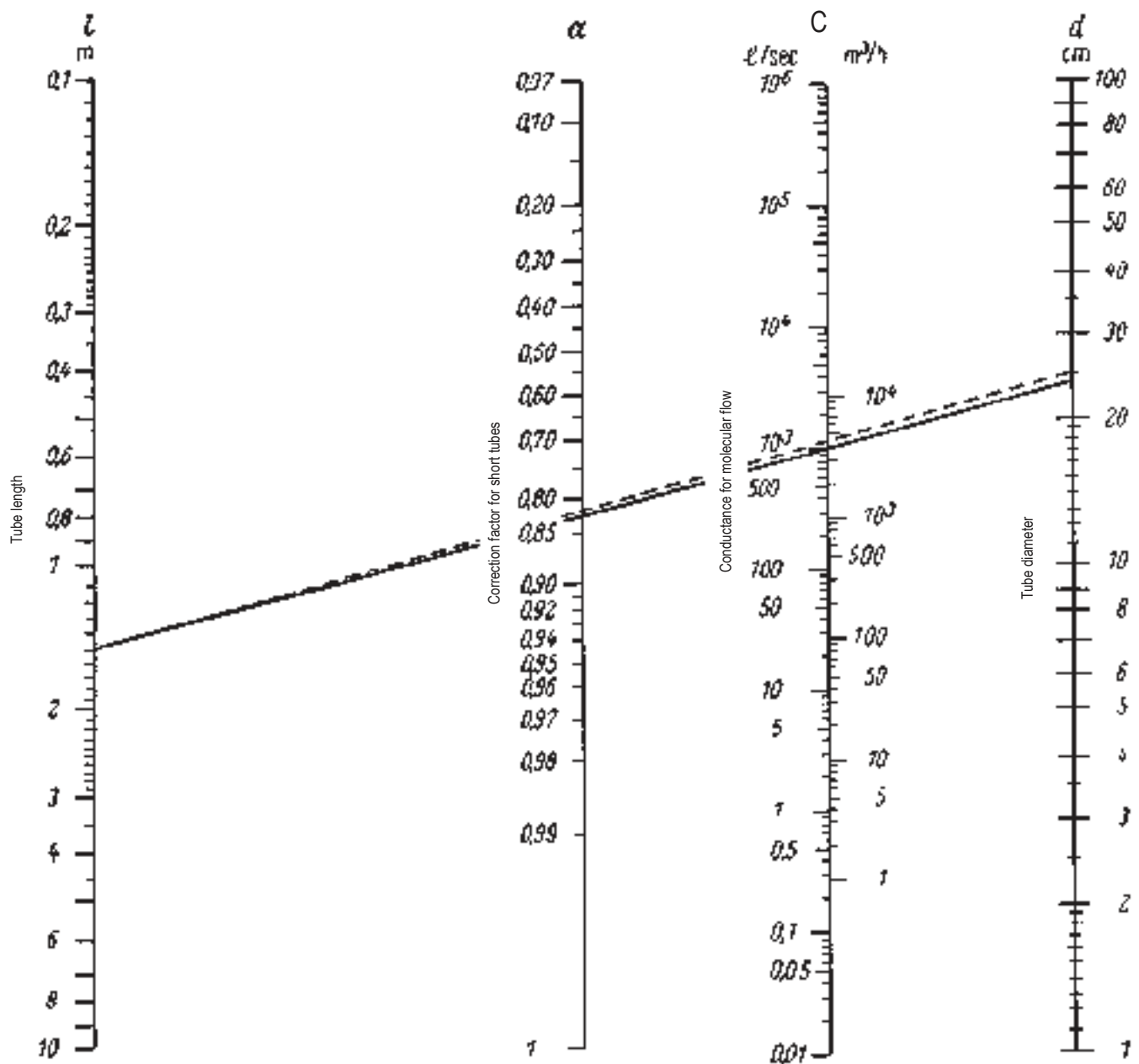
- 1) Determination of τ : A straight line is drawn through $V = 2000$ l (column ①) and $S_{eff} = 60$ m³/h = 16.7 l · s⁻¹ (column ②) and the value $t = 120$ s = 2 min is read off at the intersection of these straight lines with column ③ (note that the uncertainty of this procedure is around $\Delta t = \pm 10$ 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) $\tau = 120$ 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 t_p 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 ($S_{eff,max} = 60$ m³/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 $S_{eff,max} = 200$ m³/h = 55 l · 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 $\tau = 120$ s = 2 min from the nomogram as in example 1 (straight line through $V = 2000$ l, $S_{eff} = 16.7$ l · 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 - $4 \cdot 10^{-3}$ mbar) = $20/6 \cdot 10^{-3}$ mbar = 3300.

The time constant is obtained (straight line $V = 2000$ l in column ①, $S_{eff} = 55$ l · 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 $t_U = 1$ min for the changeover time, this results in a pump-down time of $t_p = t_{p,1} + t_U + t_{p,2} = 7.7$ min + 1 min + 4.8 min = 13.5 min.

Fig. 9.7: Nomogram for determination of pump-down time t_p 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 $\alpha = 0.83$ (intersection point of the straight line with the α 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).

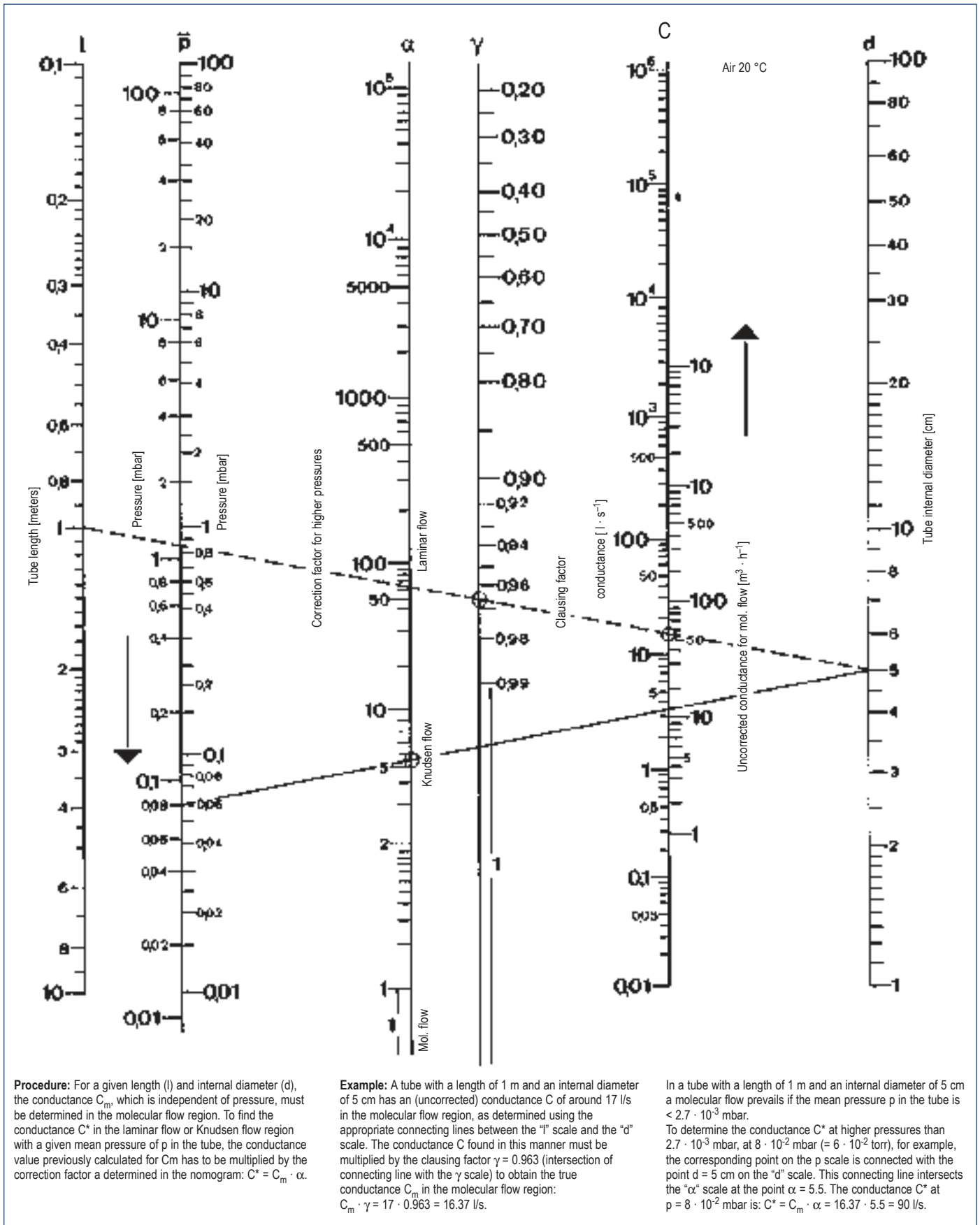
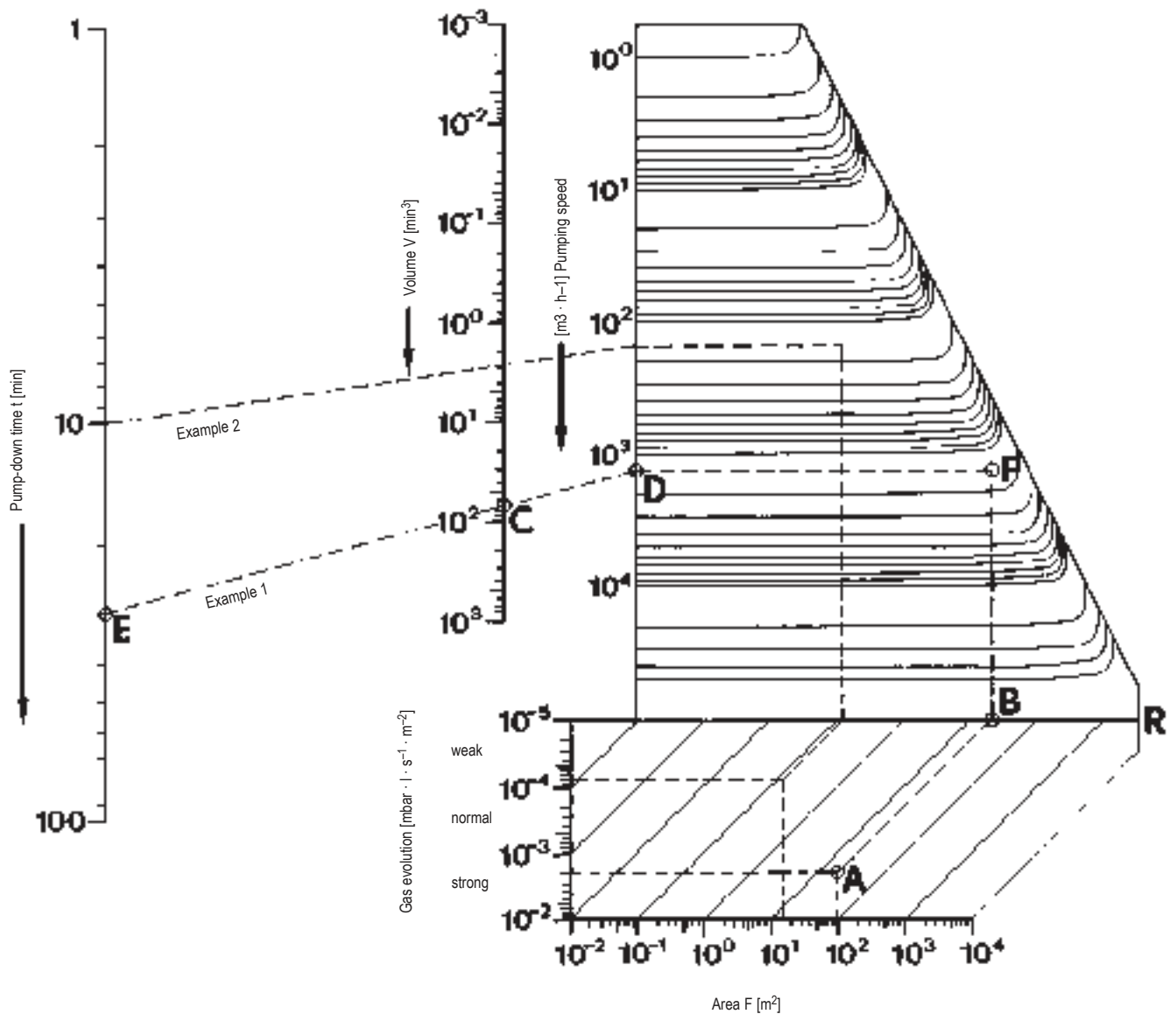


Fig. 9.9: Nomogram for determination of conductance of tubes (air, 20 °C) in the entire pressure range.



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 $2 \cdot 10^{-3} \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ is assumed. The first question is to decide whether a pump with a nominal pumping speed of $1300 \text{ m}^3/\text{h}$ is generally suitable in this case. The coordinates for the surface area concerned of 100 m^2 and a gas evolution of $2 \cdot 10^{-3} \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ 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 $1300 \text{ m}^3/\text{h}$ (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 $1300 \text{ m}^3/\text{h}$ 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

$8 \cdot 10^{-5} \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ 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 $150 \text{ m}^3/\text{h}$ is appropriate.

Fig. 9.10: Determination of pump-down time in the medium vacuum range taking into account the outgassing from the walls

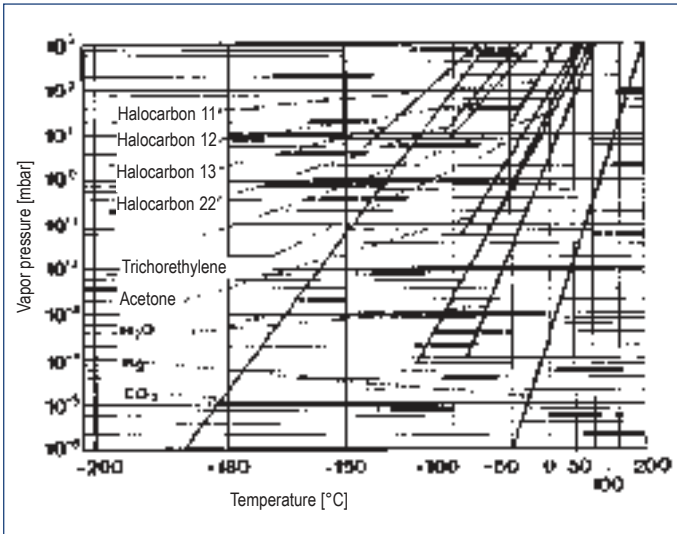


Fig. 9.11: Saturation vapor pressure of various substances

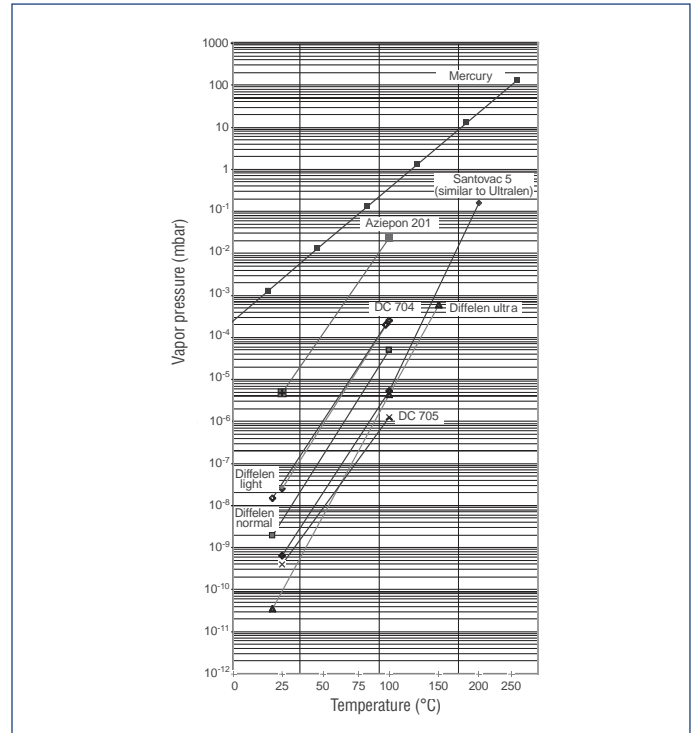


Fig. 9.12: Saturation vapor pressure of pump fluids for oil and mercury fluid entrainment pumps

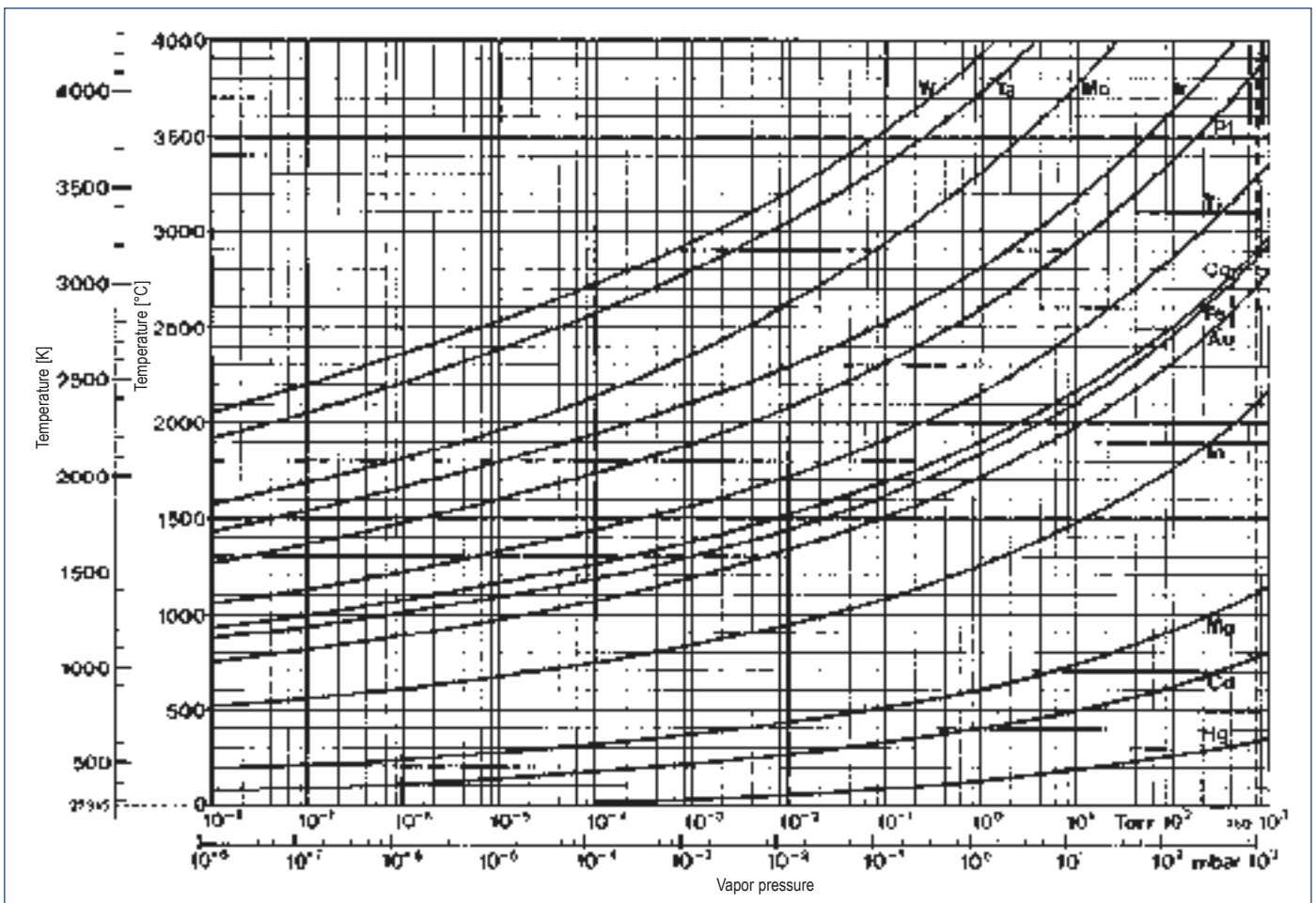


Fig. 9.13: Saturation vapor pressure of major metals used in vacuum technology

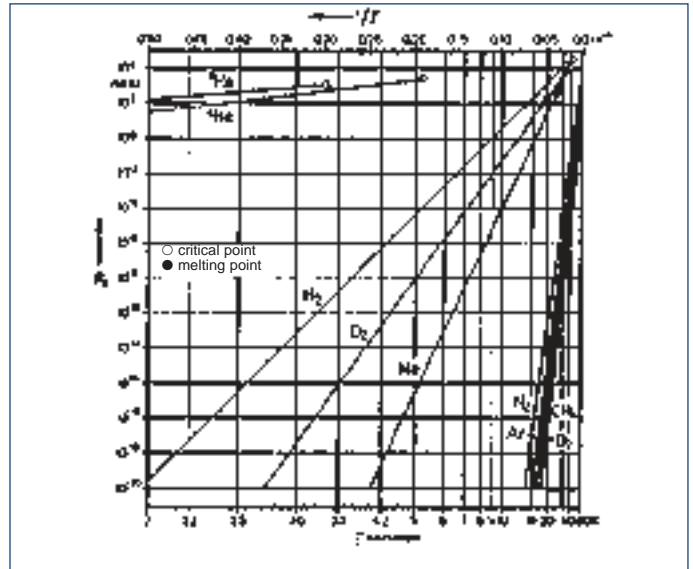
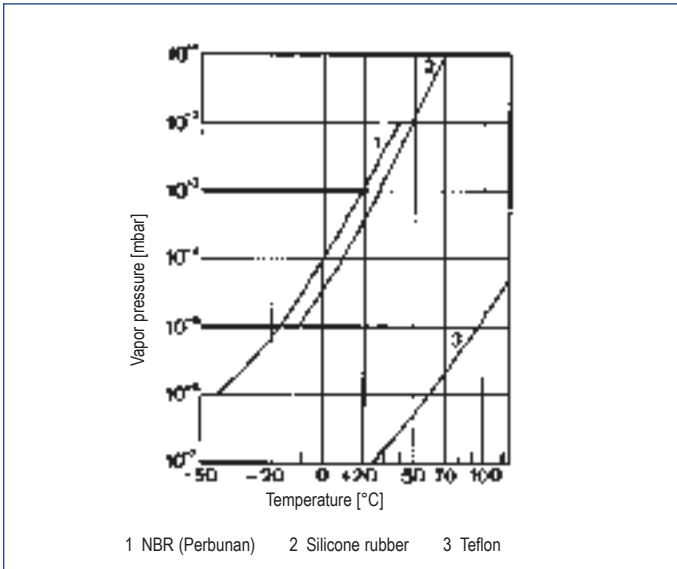


Fig. 9.14: Vapor pressure of nonmetallic sealing materials (the vapor pressure curve for fluoro rubber lies between the curves for silicone rubber and Teflon).

Fig. 9.15: Saturation vapor pressure p_s of various substances relevant for cryogenic technology in a temperaturerange of $T = 2 - 80$ K.

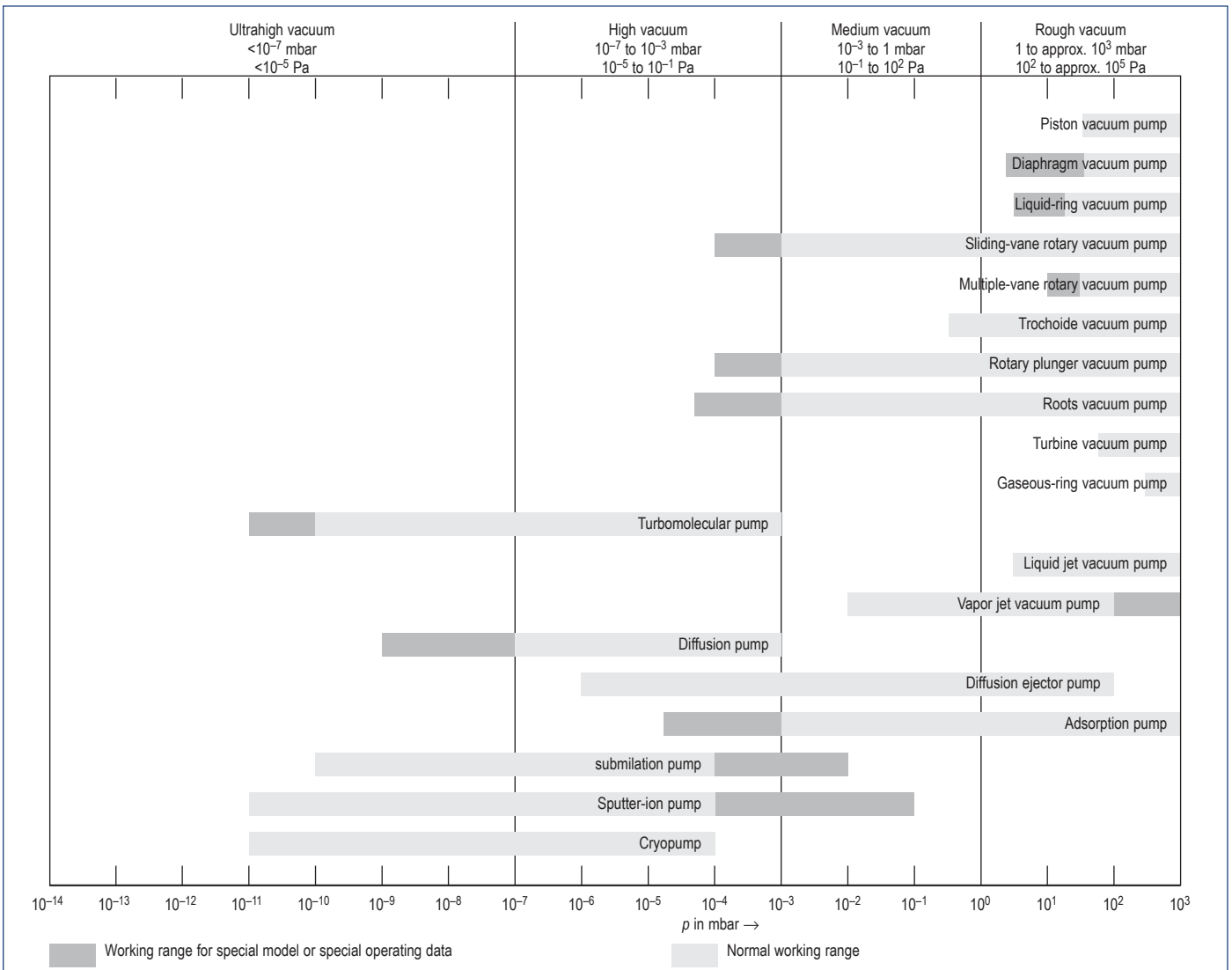


Fig. 9.16: Common working ranges of vacuum pumps

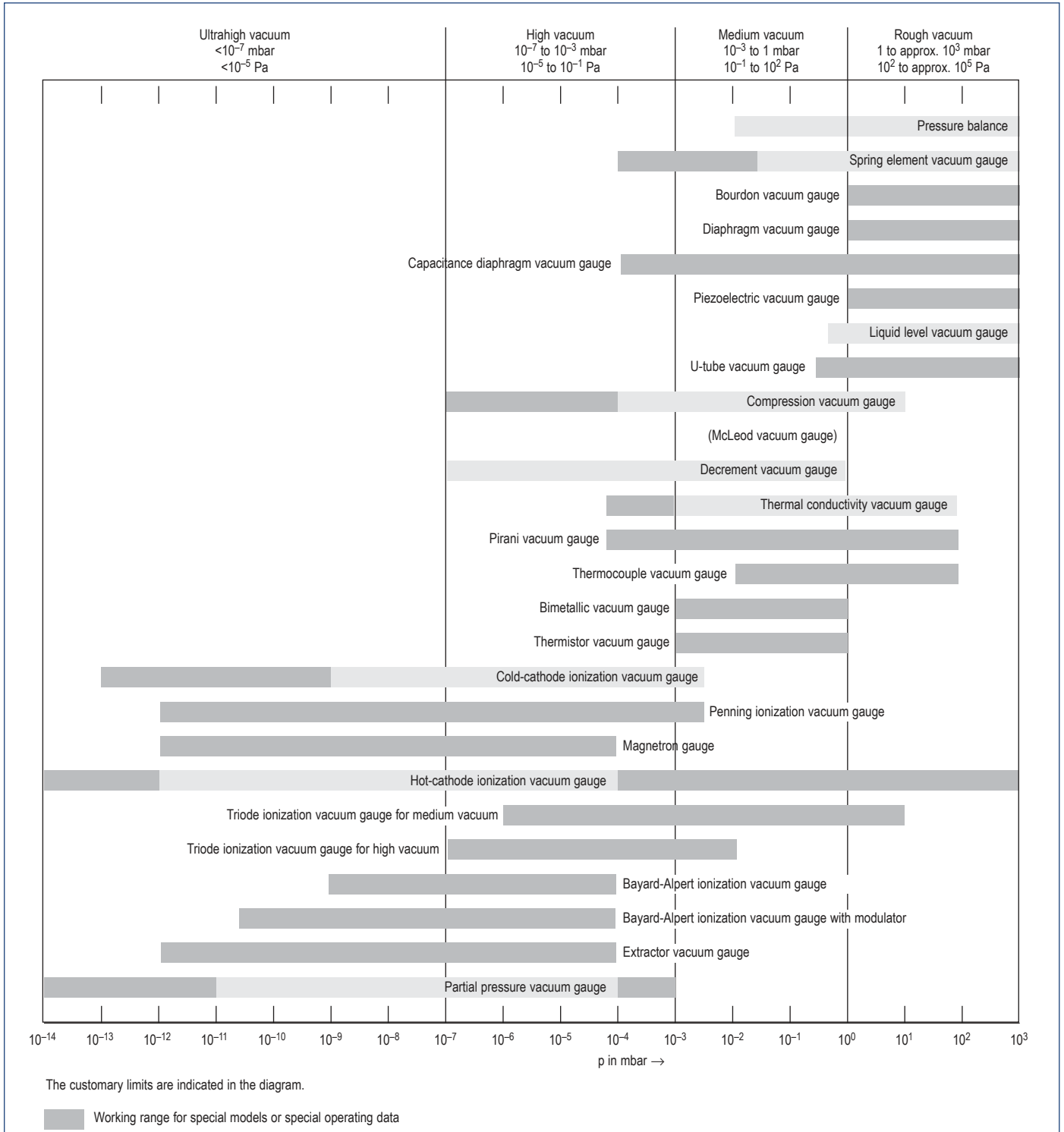


Fig. 9.16a: Measurement ranges of common vacuum gauges

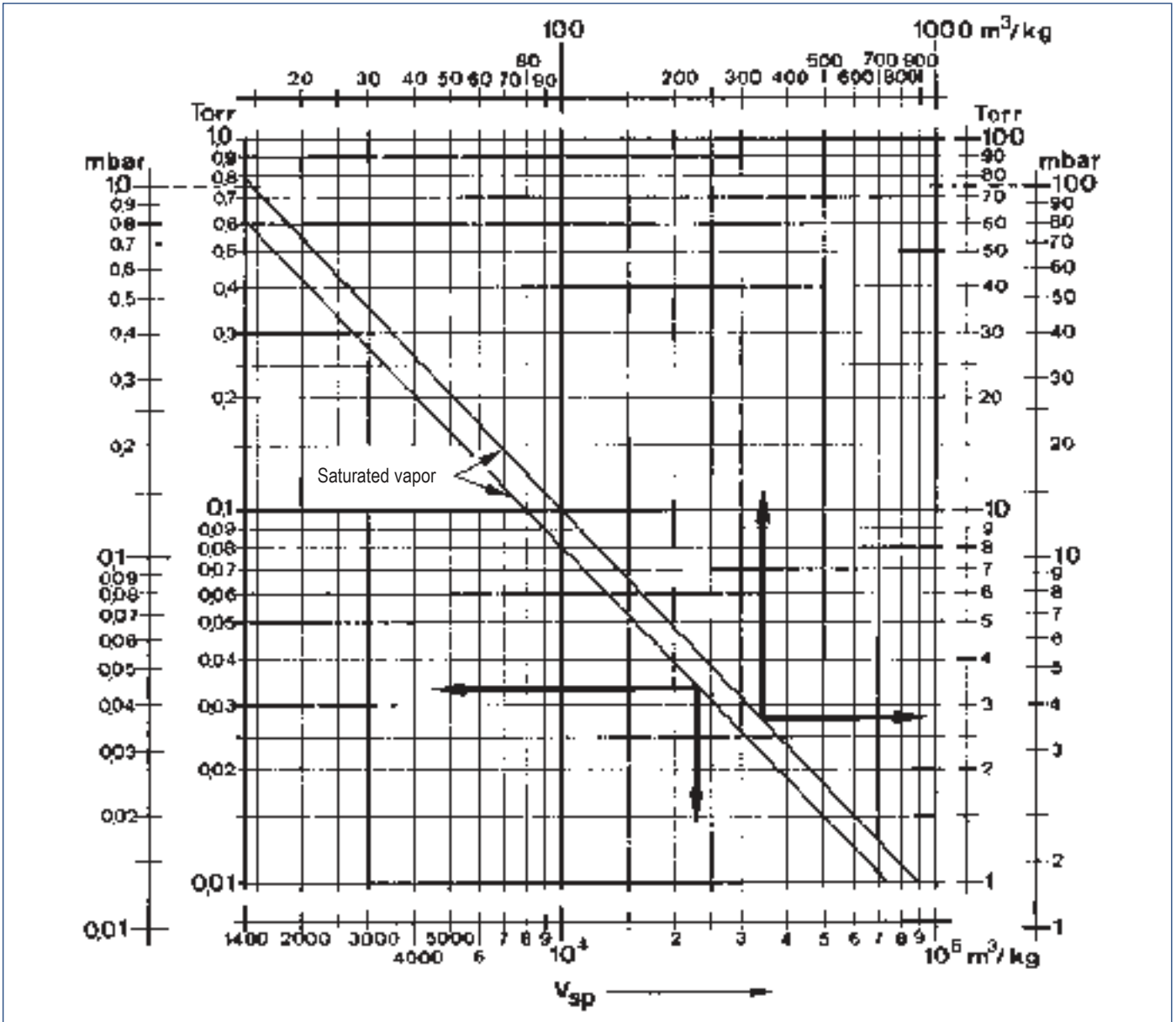


Fig. 9.17: Specific volume V_{sp} of saturated water vapor in m^3/kg within a range of 0.013 to 133 mbar.

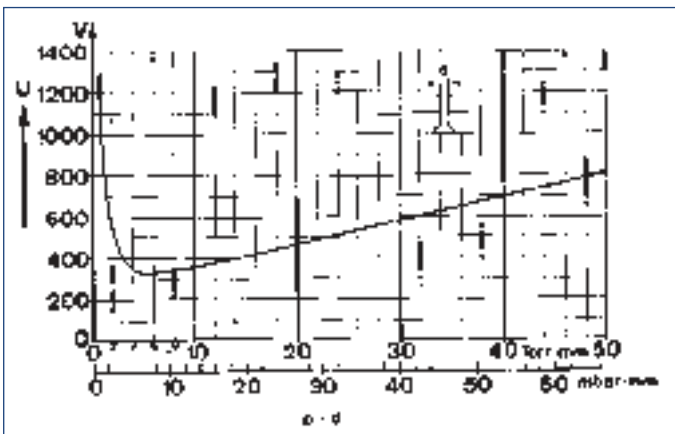


Fig. 9.18: Breakdown voltage U between parallel electrodes in a homogeneous electrical field as a function of gas pressure p distance between electrodes d (in mm) (Paschen curve), for air.

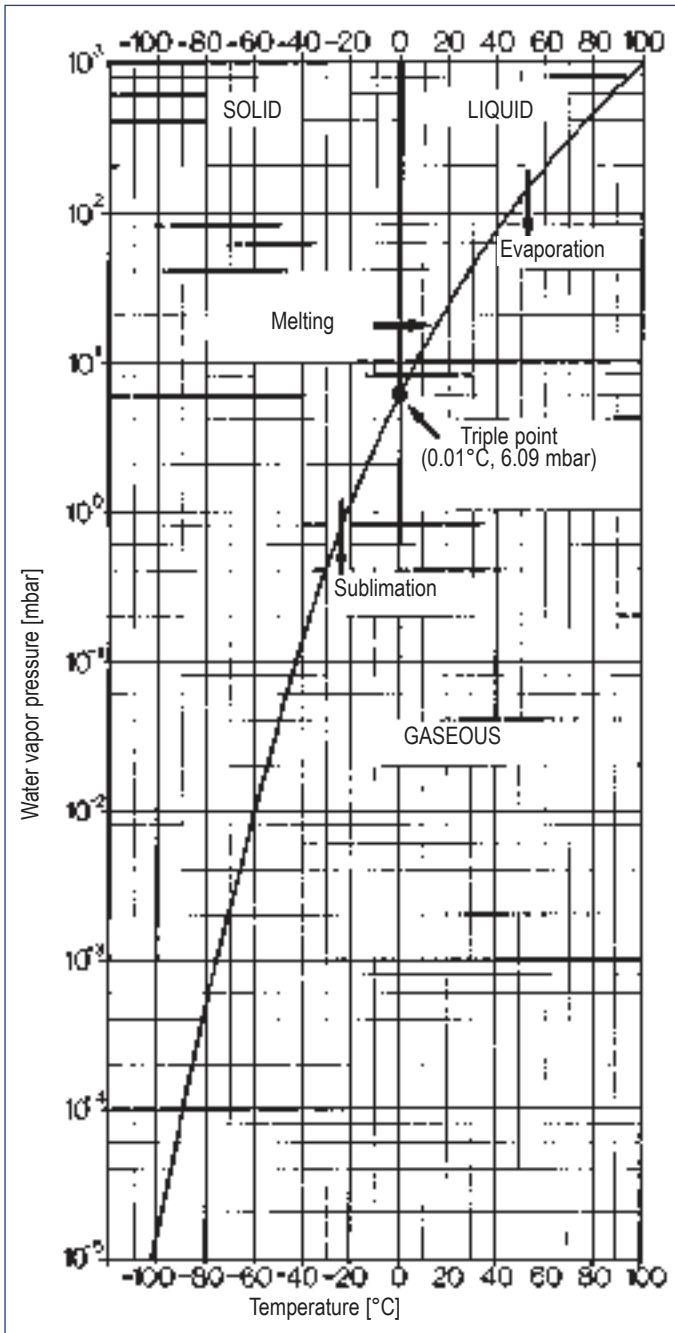


Fig. 9.19: Phase diagram of water

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:

- the basic SI units (Table 10.4.1)
- units derived from the basic SI units, in some cases with special names and unit symbols (Tables 10.4.2 and 10.4.4)
- units used in atomic physics (Table 10.4.3)
- decimal multiples and decimal parts of units, some with special names

Examples: $10^5 \text{ N} \cdot \text{m}^{-2} = 1 \text{ bar}$
 $1 \text{ dm}^3 = 1 \text{ l (liter)}$
 $10^3 \text{ kg} = 1 \text{ t (ton)}$

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 list¹ of variables, symbols and units frequently used in vacuum technology and its applications (see also DIN 28 402)

¹ The list is based on work done by Prof. Dr. I. Lückert, for which we would like to express our gratitude.

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 · s ⁻²	m · s ⁻² , cm · s ⁻²		
7	Boltzmann constant	k	J · K ⁻¹	j · K ⁻¹ , mbar · l · K ⁻¹		see Table V in Sect. 9
8	Celsius temperature	θ (theta)	–	°C	3/2	
9	Vapor pressure	p _v	N · 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 · m ⁻³	kg · m ⁻³ , g · cm ⁻³	3/6	
12	Dielectric constant	ε (epsilon)	F · m ⁻¹	F · m ⁻¹ , As · V ⁻¹ · m ⁻¹		F = Farad
13	Diffusion coefficient	D	m ² · s ⁻¹	m ² · s ⁻¹ , cm ² · s ⁻¹		
14	Moment of momentum	L	N · s · m	N · s · m		
15	Torque	M	N · m	N · m, kN · m		
16	Rotational speed, rotational frequency	n, f	s ⁻¹	s ⁻¹ , min ⁻¹		
17	Pressure in fluids	p	N · m ⁻² , Pa	bar, mbar	3/3	Pa = Pascal

No.	Variable	Symbol	SI-unit	Preferred statutory units	No. of remark in Section 10.3	Notes
18	Pressure as mechanical stress	p	N · m ⁻² , Pa	N · mm ⁻²	3/4	
19	Diameter	d	m	cm, mm		
20	Dynamic viscosity	η (eta)	Pa · s	mPa · s	3/5	
21	Effective pressure	p _e	N · m ⁻² , Pa	mbar	3/3	see also no. 126
22	Electric field strength	E	V · m ⁻¹	V · m ⁻¹		
23	Electrical capacitance	C	F	F, μF, pF		F = Farad
24	Electrical conductivity	σ (sigma)	S · m ⁻¹	S · m ⁻¹		
25	Electrical conductance	G	S	S		S = Siemens
26	Electrical voltage	U	V	V, mV, kV		
27	Electric current density	S	A · m ⁻²	a · m ⁻² , A · 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	m _e	kg	kg, g		see Table V in Sect. 9
32	Elementary charge	e	C	C, As		
33	Ultimate pressure	p _{uit}	N · m ⁻² , Pa	mbar		
34	Energy	E	J	J, kJ, kWh, eV		J = Joule
35	Energy dose	D	J · k ⁻¹		3/5 a	
36	Acceleration of free fall	g	m · s ⁻²	m · 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 · s ⁻¹	m ⁻² · s ⁻¹ ; cm ⁻² · s ⁻¹		
39	Frequency	f	Hz	Hz, kHz, MHz		
40	Gas permeability	Q _{perm}	$\frac{\text{m}^3 \text{ (NTP)}}{\text{m}^2 \cdot \text{s} \cdot \text{Pa}}$	$\frac{\text{cm}^3 \text{ (NTP)}}{\text{m}^2 \cdot \text{d} \cdot \text{bar}}$	3/19	d = day (see Tab. 10.4.4 see no. 73 and no. 103)
41	Gas constant	R				
42	Velocity	v	m · s ⁻¹	m · s ⁻¹ , mm · s ⁻¹ , km · 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 · kg ⁻¹	c · kg ⁻¹ , C · g ⁻¹	3/8	
48	Pulse	β̂ (b)	N · s	N · s		
49	Inductance	L	H	H, mH		H = Henry
50	Isoentropic exponent	κ (kappa)	–	–		κ = c _p · c _v ⁻¹
51	Isobaric molar heat capacity	C _{mp}	J · mol ⁻¹ · K ⁻¹	J · mol ⁻¹ · K ⁻¹		
52	Isobaric specific heat capacity	c _p	J · kg ⁻¹ · K ⁻¹	J · kg ⁻¹ · K ⁻¹		
53	Isochore molar heat capacity	C _{mv}	J · mol ⁻¹ · K ⁻¹			
54	Isochore specific heat capacity	c _v	J · kg ⁻¹ · K ⁻¹	J · kg ⁻¹ · K ⁻¹		
55	Kinematic viscosity	ν (nü)	m ² · s ⁻¹	mm ² · s ⁻¹ , cm ² · 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)	$\frac{\text{m}}{\text{m} \cdot \text{K}}$	$\frac{\text{m}}{\text{m} \cdot \text{K}}$; K ⁻¹		
60	Leak rate	Q _L	N · m · s ⁻¹	$\frac{\text{mbar} \cdot \text{l}}{\text{s}}$; $\frac{\text{cm}^3 \text{ (NTP)}}{\text{s}}$	3/12	
61	Power	P	W	W, kW, mW		
62	Magnetic field strength	H	A · m ⁻¹	A · m ⁻¹	3/13	
63	Magnetic flux density	B	T	T	3/14	T = Tesla
64	Magnetic flux	Φ (phi)	Wb, V · s	V · 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 · s ⁻¹	kg · s ⁻¹ , kg · h ⁻¹ , g · s ⁻¹		

No.	Variable	Symbol	SI-unit	Preferred statutory units	No. of remark in Section 10.3	Notes
68	Mass content	w_i	$\text{kg} \cdot \text{kg}^{-1}$	%, ‰, ppm		ppm = parts per million
69	Mass concentration	ρ_i (ro-i)	$\text{kg} \cdot \text{m}^{-3}$	$\text{kg} \cdot \text{m}^{-3}$, $\text{g} \cdot \text{m}^{-3}$, $\text{g} \cdot \text{cm}^{-3}$		
70	Moment of inertia	J	$\text{kg} \cdot \text{m}^2$	$\text{kg} \cdot \text{m}^2$		
71	Mean free path	λ	m	m, cm		
72	Molality	b_i	$\text{mol} \cdot \text{kg}^{-1}$	$\text{mol} \cdot \text{kg}^{-1}$		
73	Molar gas constant	R	$\frac{\text{J}}{\text{mol} \cdot \text{K}}$	$\frac{\text{mbar} \cdot \text{l}}{\text{mol} \cdot \text{K}}$		see Table V in Sect. 9
74	Molar mass (quantity-related mass)	M	$\text{kg} \cdot \text{mol}^{-1}$	$\text{kg} \cdot \text{kmol}^{-1}$, $\text{g} \cdot \text{mol}^{-1}$		
75	Molar volume	V_m	$\text{m}^3 \cdot \text{mol}^{-1}$	$\text{m}^3 \cdot \text{mol}^{-1}$, $\text{l} \cdot \text{mol}^{-1}$		
76	Molar volume, standard	V_{mn}	$\text{m}^3 \cdot \text{mol}^{-1}$	$\text{m}^3 \cdot \text{mol}^{-1}$ (NTP) $\text{l} \cdot \text{mol}^{-1}$ (NTP)		see Table V in Sect. 9
77	Molecular mass	m	kg	g		
78	Normal stress (mech.)	σ (sigma)	$\text{N} \cdot \text{m}^{-2}$	$\text{N} \cdot \text{mm}^{-2}$		
79	Standard density of a gas	ρ_n (ro-en)	$\text{kg} \cdot \text{m}^{-3}$	$\text{kg} \cdot \text{m}^{-3}$, $\text{g} \cdot \text{cm}^{-3}$		
80	Standard pressure	p_n	$\text{N} \cdot \text{m}^{-2}$, 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	$\text{N} \cdot \text{m}^{-2}$, Pa	mbar	3/17	
83	Period	T	s	s, ms, μs		
84	Permeation coefficient	P	$\frac{\text{m}^3 \cdot \text{m}}{\text{s} \cdot \text{m}^2 \cdot \text{bar}}$	$\frac{\text{cm}^2}{\text{s} \cdot \text{mbar}}$	3/18	
85	Planck constant	h	$\text{J} \cdot \text{s}$	$\text{J} \cdot \text{s}$		see Table V in Sect. 9
86	pV throughput	q_{pV}	$\text{N} \cdot \text{m} \cdot \text{s}^{-1}$	$\text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$	3/19	
87	pV value	pV	$\text{N} \cdot \text{m}$	$\text{mbar} \cdot \text{l}$	3/19	
88	Radius (also molecular radius)	r	m	cm, mm, μm		
89	Space charge density	ρ (ro)	$\text{C} \cdot \text{m}^{-3}$	$\text{C} \cdot \text{m}^{-3}$, $\text{As} \cdot \text{m}^{-3}$		
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}	$\text{N} \cdot \text{m}^{-2}$, Pa	mbar		
95	Residual gas pressure	p_{rg}	$\text{N} \cdot \text{m}^{-2}$, Pa	mbar		
96	Residual (total) pressure	p_r	$\text{N} \cdot \text{m}^{-2}$, Pa	mbar		
97	Reynold number nondimensional variable	Re	–	–		nondimensional variab.
98	Saturation vapor pressure	p_s	$\text{N} \cdot \text{m}^{-2}$, Pa	mbar		
99	Throughput (of a pump)	q_{pV} , Q	$\text{N} \cdot \text{m} \cdot \text{s}^{-1}$	$\text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$		
100	Pumping speed	S	$\text{m}^3 \cdot \text{s}^{-1}$	$\text{m}^3 \cdot \text{h}^{-1}$, $\text{l} \cdot \text{s}^{-1}$		see no. 132
101	Stress (mech.)	ρ , σ , τ (ro, sigma, tau)	$\text{N} \cdot \text{m}^{-2}$	$\text{N} \cdot \text{m}^{-2}$, $\text{N} \cdot \text{mm}^{-2}$	3/4	see no. 18
102	Specific electron charge	$-e \cdot m_e^{-1}$	$\text{C} \cdot \text{kg}^{-1}$	$\text{C} \cdot \text{kg}^{-1}$, $\text{As} \cdot \text{kg}^{-1}$		see Table V in Sect. 9
103	Specific gas constant	R_i	$\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$	$\frac{\text{mbar} \cdot \text{l}}{\text{kg} \cdot \text{K}}$	3/22	
104	Specific ion charge	$e \cdot m^{-1}$	$\text{C} \cdot \text{kg}^{-1}$	$\text{C} \cdot \text{kg}^{-1}$, $\text{As} \cdot \text{kg}^{-1}$		
105	Specific electrical resistance	ρ (ro)	$\Omega \cdot \text{m}$	$\Omega \cdot \text{cm}$, $\Omega \cdot \text{mm}^2 \cdot \text{m}^{-1}$		
106	Specific volume	v	$\text{m}^3 \cdot \text{kg}^{-1}$	$\text{m}^3 \cdot \text{kg}^{-1}$; $\text{cm}^3 \cdot \text{g}^{-1}$		
107	Specific heat capacity	c	$\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$	$\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$, $\text{J} \cdot \text{g}^{-1} \cdot \text{K}^{-1}$	3/23	
108	Stefan-Boltzmann constant	σ (sigma)	$\frac{\text{W}}{\text{m}^2 \cdot \text{K}^4}$	$\frac{\text{W}}{\text{m}^2 \cdot \text{K}^4}$		see Table V in Sect. 9
109	Quantity of substance	v (n \ddot{u})	mol	mol, kmol		
110	Throughput of substance	q_v	$\text{mol} \cdot \text{s}^{-1}$	$\text{mol} \cdot \text{s}^{-1}$		
111	Concentration of substance	c_i	$\text{mol} \cdot \text{m}^{-3}$	$\text{mol} \cdot \text{m}^{-3}$, $\text{mol} \cdot \text{l}^{-1}$		for substance „i“
112	Collision rate	Z	s^{-1}	s^{-1}		

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	$\Delta T, \Delta \vartheta$	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)	$m^3 \cdot m^{-3}$	$l \cdot l^{-1}, \%, \text{‰}, ppm$		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).

$$1 \text{ Ci} = 3.7 \cdot 10^{10} \cdot \text{s}^{-1} = 37 \text{ ns}^{-1}$$

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 · 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 = 10² 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 : $p_e = p - p_{\text{amb}}$. The overpressure can have positive or negative values.

Conversions

$$1 \text{ kg} \cdot \text{cm}^{-2} = 980.665 \text{ mbar} = \mathbf{981 \text{ mbar}}$$

$$1 \text{ at (technical atmosphere)} = 980.665 \text{ mbar} = \mathbf{981 \text{ mbar}}$$

$$1 \text{ atm (physical atmosphere)} = 1013.25 \text{ mbar} = 1013 \text{ mbar}$$

$$1 \text{ atmosphere above atmospheric pressure (atmospheric overpressure)} = 2026.50 \text{ mbar} = 2 \text{ bar}$$

$$1 \text{ torr} = 1 \text{ mm Hg} = \frac{1 \text{ atm}}{760} = 133.322 \text{ Pa} = 1.333 \text{ mbar}$$

$$1 \text{ meter head of water} = 9806.65 \text{ Pa} = 98 \text{ mbar}$$

$$1 \text{ mm Hg} = 133.332 \text{ Pa} = 1.333 \text{ mbar} = 4/3 \text{ mbar}$$

The pressure as mechanical stress (strength) is generally given in pascal (Pa) and in N · mm⁻².

Conversions:

$$1 \text{ Pa} = 1 \text{ N} \cdot \text{m}^{-2} = 10^{-6} \text{ N} \cdot \text{mm}^{-2}$$

$$1 \text{ kg} \cdot \text{cm}^{-2} = 98,100 \text{ Pa} = 0.981 \text{ N} \cdot \text{mm}^{-2} = 0,1 \text{ N mm}^{-2}$$

$$1 \text{ kg} \cdot \text{mm}^{-2} = 9,810,000 \text{ Pa} = 9.81 \text{ N} \cdot \text{mm}^{-2} = 10 \text{ N} \cdot \text{mm}^{-2}$$

3/5: Dynamic viscosity

The unit previously used was poise (P).

$$1 \text{ P} = 0.1 \text{ Pa} \cdot \text{s} = 1 \text{ g} \cdot \text{cm}^{-1} \cdot \text{s}^{-1}$$

3/5a: Energy dose

Rad (rd) is no longer permissible.

$$1 \text{ rd} = \frac{1}{100} \text{ J} \cdot \text{kg}^{-1}$$

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.

$$\mathbf{1 \text{ kp} = 9.81 \text{ N}}$$

3/8: Ion dose

The previously used unit was the Röntgen (R). $1 \text{ R} = 2,58 \cdot 10^{-4} \text{ C} \cdot \text{kg}^{-1}$

3/9: Kinematic viscosity

The previously used unit was stokes (St).

$$1 \text{ St} = 1 \text{ cm}^2 \cdot \text{s}^{-1}; 1 \text{ cSt} = 1 \text{ mm}^2 \cdot \text{s}^{-1}$$

3/10: Force

The dyne, the CGS unit for force, is no longer used.

$$1 \text{ dyne} = 10^{-5} \text{ N}$$

3/11: Length/wavelength

The unit Ångström (Å) (e.g. for wavelength) will no longer be used in future (see Table 4.6).

$$1 \text{ Å} = 10^{-8} \text{ cm} = 0.1 \text{ nm}$$

3/12: Leak rate

In DIN 40.046 sheet 102 (draft of August 1973 issue), the unit $\text{mbar} \cdot \text{dm}^3 \cdot \text{s}^{-1}$ (= $\text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$) is used for the leak rate. Note that the leak rate corresponding to the unit $1 \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1}$ at 20 °C is practically the same as the leak rate $1 \text{ cm}^3 \cdot \text{s}^{-1}$ (NTP). (See also 3/17)

3/13: Magnetic field strength

The previously used unit was the oersted (Oe).

$$1 \text{ Oe} = 79.577 \text{ A} \cdot \text{m}^{-1}$$

3/14: Magnetic flux density

The previously used unit was the gauss (G).

$$1 \text{ G} = 10^{-4} \text{ Vs} \cdot \text{m}^{-2} = 10^{-4} \text{ T (T = Tesla)}$$

3/15: Magnetic flux

The previously used unit was the maxwell (M).

$$1 \text{ M} = 10^{-8} \text{ Wb (Weber)}$$

3/16: Standard volume

DIN 1343 must be complied with.

The designation m^3 (NTP) or $\text{m}^3 (p_n, T_n)$ 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 ($T_n = 273 \text{ K}$, $p_n = 1013 \text{ mbar}$).

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 $\text{m}^3 \cdot \text{s}^{-1}$ (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". $R_i = R_m (M_i^{-1}; M_i$ 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.

$$1 \text{ kcal} = 4.2 \text{ kJ}$$

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).

$$1^\circ = \frac{\pi}{180} \text{ rad}; 1' = 1^\circ/60; 1'' = 1'/60.$$

$$1 \text{ rad} = \frac{360^\circ}{2\pi} \cdot 60^\circ$$

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 coherent¹ SI units with special names and symbols (alphabetical)

Name of unit	Symbol	Variable	Relationship
coulomb	C	quantity of electricity or electric charge	$1 \text{ C} = 1 \text{ A} \cdot \text{s}$
farad	F	electrical capacitance	$1 \text{ F} = 1 \text{ A} \cdot \text{s} \cdot \text{V}^{-1}$
henry	H	inductance	$1 \text{ H} = 1 \text{ V} \cdot \text{s} \cdot \text{A}^{-1}$
hertz	Hz	frequency	$1 \text{ Hz} = 1 \cdot \text{s}^{-1}$
joule	J	energy, work, quantity of heat	$1 \text{ J} = 1 \text{ N} \cdot \text{m} = \text{Ws}$
lumen	lm	luminous flux	$1 \text{ lm} = \text{cd} \cdot \text{sr}$
lux	lx	illuminance	$1 \text{ lx} = 1 \text{ lm} \cdot \text{m}^{-2}$
newton	N	force	$1 \text{ N} = 1 \text{ kgm} \cdot \text{s}^{-2}$
ohm	Ω	electrical resistance	$1 \Omega = 1 \text{ V} \cdot \text{A}^{-1}$
pascal	Pa	pressure, mechanical stress	$1 \text{ Pa} = 1 \text{ N} \cdot \text{m}^{-2}$
radian	rad ²	angle, plane angle	$1 \text{ rad} = 1 \text{ m} \cdot \text{m}^{-1}$
siemens	S	electrical conductance	$1 \text{ S} = 1 \cdot \Omega^{-1}$
steradian	sr ²	solid angle	$1 \text{ sr} = 1 \text{ m}^2 \cdot \text{m}^{-2}$
tesla	T	magnetic flux density or induction	$1 \text{ T} = 1 \text{ Wb} \cdot \text{m}^{-2}$
volt	V	electrical voltage or potential difference	$1 \text{ V} = 1 \text{ W} \cdot \text{A}^{-1}$
watt	W	power, energy flux, heat flux	$1 \text{ W} = 1 \text{ J} \cdot \text{s}^{-1}$
weber	Wb	magnetic flux	$1 \text{ Wb} = 1 \text{ V} \cdot \text{s}$

¹ Formed with numeric factor 1; e.g. $1 \text{ C} = 1 \text{ As}$, $1 \text{ Pa} = 1 \text{ N} \cdot \text{m}^{-2}$

² Additional SI unit

10.4.4 Derived noncoherent SI units with special names and symbols

Basic unit	Symbol	Definition
Day	d	$1 \text{ d} = 86.400 \text{ s}$
Hour	h	$1 \text{ h} = 3.600 \text{ s}$
Minute	min	$1 \text{ min} = 60 \text{ s}$
Round angle	–	$2 \pi \text{ rad}$
Degree	(°)	$\frac{\pi}{180} \text{ rad}$
Minute	(')	$\frac{\pi}{10.800} \text{ rad} (= \frac{1}{60} \text{ grad})$
Second	(")	$\frac{\pi}{648.000} \text{ rad} (= \frac{1}{60} \text{ minute})$

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

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	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

DIN	Title	Issue	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)	28410	Vacuum technology; mass spectrometer partial pressure gauges, definitions, characteristic variables, operating conditions	11/86
16005	Overpressure gauges with elastic measuring element for general use Requirements and testing	2/87	28411	Vacuum technology; acceptance specifications for mass spectrometer leak detection devices, definitions	3/76
16006	Overpressure gauges with Bourdon tube Safety-related requirements and testing	2/87	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
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	28417	Vacuum technology; measuring pV mass flow according to volumetric method at constant pressure	3/76
25436	Integral leak rate test of safety vessel with absolute pressure method	7/80	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
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	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	
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)	28427	Vacuum technology; acceptance specifications for diffusion pumps and ejector vacuum pumps for pump fluid vapor pressures of less than 1 mbar	2/83
28401	Vacuum technology; symbols – overview	11/76	28428	Vacuum technology; acceptance specifications for turbo-molecular pumps	11/78
28402	Vacuum technology: variables, symbols, units - overview	12/76	28429	Vacuum technology; acceptance specifications for getter-ion pumps	8/85
28403	Vacuum technology; quick connections, small flange connections	9/86	28430	Vacuum technology; measuring specifications for ejector vacuum pumps and ejector compressors. Pump fluid: water vapor	11/84
28404	Vacuum technology: flanges, dimensions	10/86	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

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
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1518 E	Nondestructive testing – determination of characteristic variables for mass spectrometer leak detectors	
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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

ISO	Title	Issue
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 of volume 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

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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
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