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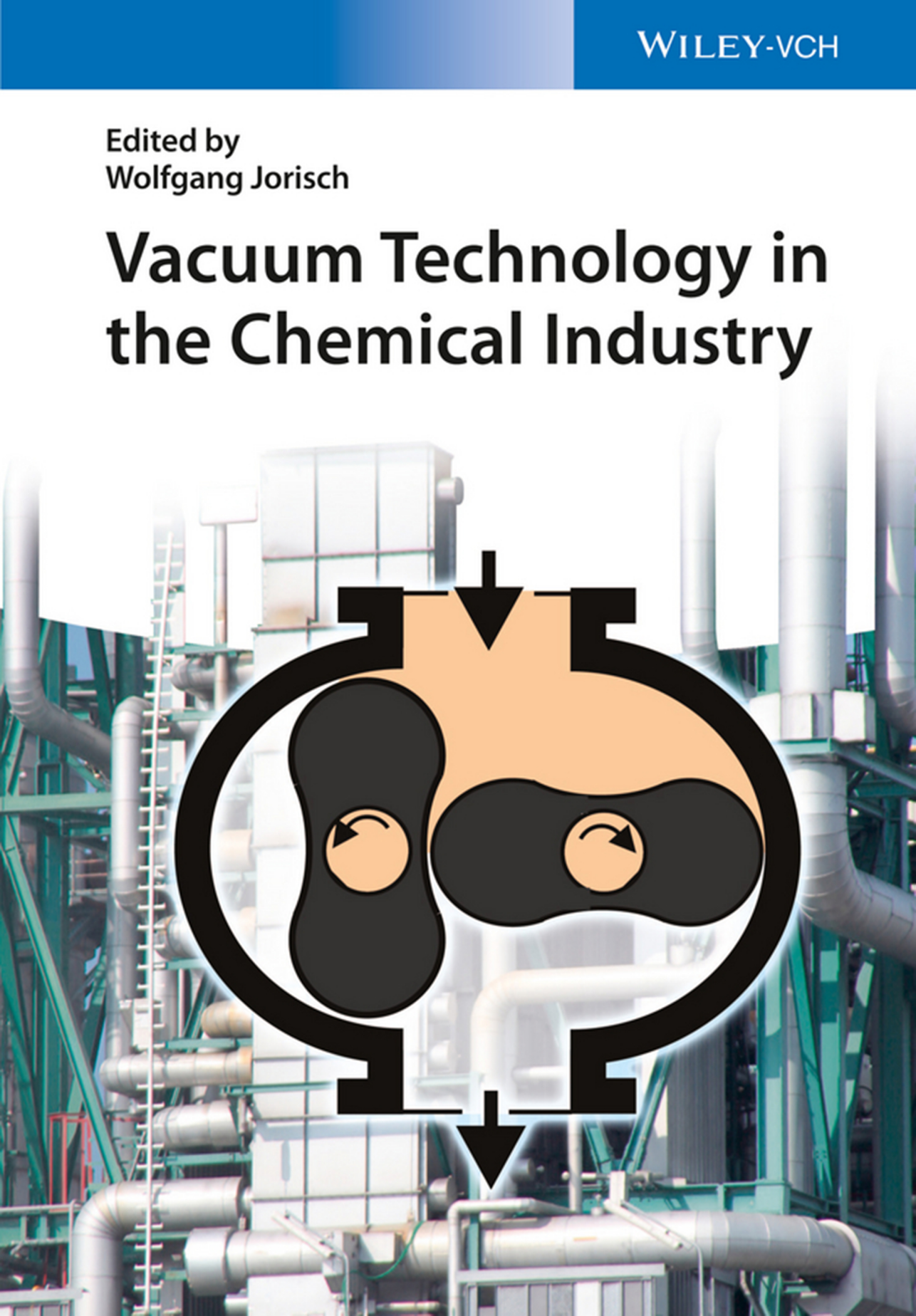

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1
Fundamentals of Vacuum Technology

Wolfgang Jorisch

1.1
Introduction

Vacuum technology is being used widely in many chemistry applications. Here it is not used in the same way as in physics applications. In physics applications, it is the objective to perform experiments in volumes (vacuum chambers) which are as pure as possible, that is, which contain as few particles as possible as these particles generally impair the physical process.

Vacuum technology is used in the area of chemistry applications for the purpose of performing basic thermal and mechanical operations to reprocess reaction products under conditions which preserve the product. Typical applications for thermal separating processes in a vacuum are distillation, drying or sublimation at reduced pressures as well as applications which accelerate the reaction itself when reaction products from the reaction mixture need to be removed for the purpose of shifting the equilibrium in the desired direction, for example. An example of this is the process of esterification.

A mechanical process performed in a vacuum is that of vacuum filtration where the pressure difference created between vacuum and atmospheric pressure is utilised as the driving force for the filtration process.

The planning process engineer or the consulting engineer of a chemical plant not only faces questions how to properly dimension a vacuum system so as to comply with the demanded process specifications, but he needs to solve in a satisfactory way, problems relating to operating costs which shall be as low as possible and questions as to the minimisation of emissions in the discharged air and waste water. The wide variety of vacuum pumps used in the area of chemistry technology reflects this. The responsible planning engineer or plant chemist will have to select, in consideration of the process engineering questions which differ from process to process, vacuum generators which promise to offer the best possible solution for the specific case.

For this reason, this book covers besides vacuum process engineering fundamentals, above all also the different types of vacuum pumps.

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1.2 Fundamentals of Vacuum Technology

Also for the vacuum technology used in chemistry applications, the underlying fundamental laws of physics apply.

The standard DIN 28400 Part 1 defines the vacuum state as

Vacuum is the state of a gas, the particle number density of which is below that of the atmosphere at the Earth's surface. Since the particle number density is within certain limits time and location dependent, it is not possible to state a general upper limit for the vacuum. Here the gas particles exert a pressure on all bodies which surround them, this pressure being the result of their temperature dependent motion. The pressure is defined as a force per unit of area, with the unit of measurement being the Pascal.

$$1\,\mathrm{Pa} = 1\,\mathrm{Nm}^{-2} \tag{1.1}$$

In the area of vacuum process engineering, frequently not Pascal is used as the unit of measurement for pressures but instead also the allowed unit 'bar' or 'mbar'.

$$1\,\mathrm{mbar} = 10^2\,\mathrm{Pa} = 1\,\mathrm{hPa} \tag{1.2}$$

Owing to the differing behaviour of the particles within the considered volume (particle number density) which is dependent on the number of particles which are present, different pressure (vacuum) ranges have been defined with respect to their flow characteristics, for example:

	Pressure range (mbar)
Rough vacuum	$< 1.013\,10^3 - 1$
Medium vacuum	$< 1 - 10^{-3}$
High vacuum	$< 10^{-3} - 10^{-7}$
Ultrahigh vacuum	$< 10^{-7}$
Remark:	
New definition of beginning rough vacuum:	
Lowest pressure on Earth surface	300 mbar (Mount Everest)

The pressure When gas particles impinge on a wall (surface) they are subjected to a change in impulse, whereby they transfer an impulse to this wall. This impulse is the cause for the pressure exerted on the wall:

$$p = \frac{d(mv)}{Adt} = \frac{F}{A} \tag{1.3}$$

since the force is equivalent to the change in impulse of over time:

$$\frac{d(mv)}{dt} \tag{1.4}$$

p = pressure; F = force; A = area; m = mass; v = velocity; and t = time.

Table 1.1 Composition of atmospheric air.

Constituent	Volume share (%)	Partial pressure (mbar)
Nitrogen	—	78.09
	—	780.9
Oxygen	—	20.95
	—	209.5
Argon	—	0.93
	—	9.3
Carbon dioxide	—	0.03
	—	3.10^{-1}
Water vapour	≤2.3	—
	≤ 23.3	—

Remainder: noble gases, hydrogen, methane, ozone, and so on.

When now considering a surface onto which particles impinge from a hemisphere and when integrating the transferred impulse over time, then one obtains for ideal gases

$$p = \frac{d(mv)}{Adt} = \overline{n}kT \tag{1.5}$$

$\overline{n}$ = particle number density; k = Boltzmann constant and T = absolute temperature.

That is, the exerted pressure is only dependent on the number of gas molecules n in the volume but is not dependent on the type of gas [1].

This statement ultimately confirms also Dalton's Law, which states that the total pressure of a gas atmosphere is equal to the sum of all partial pressures of this gas mixture:

$$p_{\text{tot}} = \overline{n}_{\text{tot}}kT = \overline{n}_1kT + \overline{n}_2kT + \cdots \overline{n}_ikT \tag{1.6}$$

or

$$p_{\text{tot}} = p_1 + p_2 + \cdots + p_i \tag{1.7}$$

p_{tot} = total pressure; $\overline{n}_{\text{tot}}$ = particle number density of all gas particles (types); and $\overline{n}_i$ = particle number density of the particle type *i.*

Given as an example is in the following the composition of air at atmospheric pressure [2] (Table 1.1).

1.2.1 Fundamentals of Gas Kinetics

The individual molecules or gas particles contained in a volume are in constant motion and collide with each other. In doing so, the particles change their velocity each time they collide. From a statistical point of view, all velocities are possible but with differing probability.

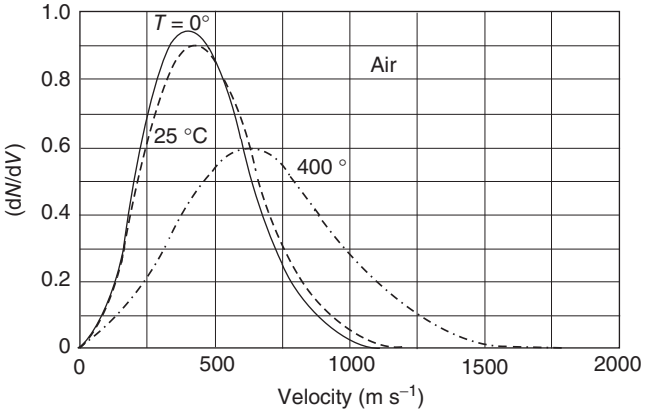

Figure 1.1 Velocity distribution of air molecules (nitrogen and oxygen) at 0, 25 and at 400 °C.

Maxwell and Boltzmann found the following relationship for the velocity distribution of the gas particles [3]:

$$\frac{dn}{dv} = \frac{2N}{\pi^{\frac{1}{2}}}\left(\frac{m}{2kT}\right)^{\frac{3}{2}} v^2 e^{\frac{-mv^2}{2kT}} \tag{1.8}$$

m	=	mass of each particle
T	=	temperature in Kelvin
N	=	number of particles
k	=	Boltzmann constant
v	=	velocity of the particles.

Figure 1.1 depicts the velocity distribution between velocity v and $v + dv$ based on the example of air at 0, 25 and 400 °C [3].

From this, it is apparent that there does not exist a molecule with a velocity of 'zero' or with an infinitely high velocity. The location of the most probable velocity (maximum, v_p) is a function of the mean gas temperature. Moreover, the molecule velocity depends on the molar mass. The most likely velocity can be stated through

$$v_p = \left(\frac{2kT}{m}\right)^{\frac{1}{2}} \tag{1.9}$$

and the arithmetic mean velocity can be stated through

$$v = \left(\frac{8kT}{\pi m}\right)^{\frac{1}{2}} \tag{1.10}$$

Figure 1.2 depicts the velocity of the gas molecules as a function of the type of gas.

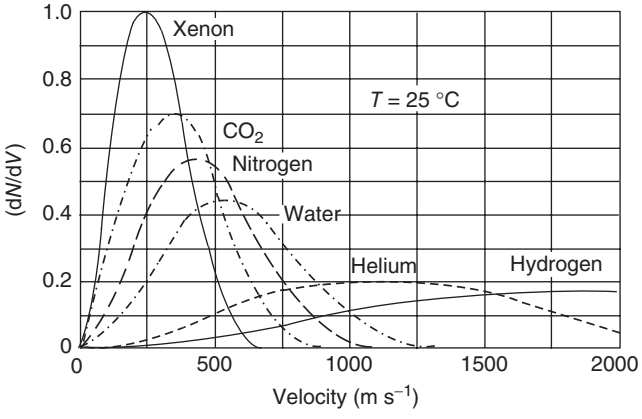

Figure 1.2 Gas molecule velocity as a function of the type of gas.

1.2.1.1 **Mean Free Path**

The fact that the molecules move at different velocities allows for the conclusion that they will move within a specific unit of time over a different distance (free path) before colliding with another particle. The mean free path λ, resulting from the kinetic gas theory is

$$\lambda = \frac{1}{2^{\frac{1}{2}}\pi d_0^2 \overline{n}} \tag{1.11}$$

where d_0 = collision radius in [mm] of an ideal point-like particle and $\overline{n}$ is the particle number density (number of molecules per volume).

Since the particle number density, as already derived, depends on the pressure, also the mean free path of the gas molecules is pressure dependent (at constant temperature), the product of the prevailing pressure and the mean free path is, at a given temperature, a constant (gas-type dependent).

For nitrogen at 20 °C, this product amounts to $\lambda \cdot p = 6.5 \cdot 10^{-5}$ mbar, that is at a prevailing pressure of $p = 1 \times 10^{-2}$ mbar there results at a temperature of 20 °C a mean free path of 6.5 mm [4].

Moreover, kinetic gas theory states the distribution of the mean free paths as follows:

$$N = N' e^{-\frac{x}{\lambda}} \tag{1.12}$$

N'	=	number of molecules in the volume
N	=	number of the molecules which have a free path of x.

From this equation, it can be derived that 63% of all collisions occur after a path of $0 \leq x \leq \lambda$ whereas 37% of the collisions occur after a path of $\lambda < x \leq 5\lambda$.

Only 0.6% have a free path exceeding 5λ.

1.2.2 Equation of State for Ideal Gases

In the year 1662, Robert Boyle demonstrated that the volume of a gas, provided it is maintained at a constant temperature, is inversely proportional to the pressure:

$$p_1 V_1 = p_2 V_2 \ (N, T \text{ constant(isotherm)}) \ Boyle's\ Law \tag{1.13}$$

In 1787, the French chemist Charles found the law according to which the volume of a gas is proportional to its temperature when maintaining it at a constant pressure:

$$\frac{V_1}{T_1} = \frac{V_0}{T_0} (N,\ p \text{ constant(isobar)}) \tag{1.14}$$

$$V_1 = V_0 \frac{273.15 + \vartheta}{273.15} \ Charles' Law$$
$$\vartheta = \text{temperature in } ^\circ\text{C} \tag{1.15}$$

Also, the French physicist Amontons discovered that the pressure of a gas is proportional to its temperature when maintaining it at a constant volume:

$$\frac{p_1}{T_1} = \frac{p_0}{T_0} (N, V \text{ constant(isochore)}) \tag{1.16}$$

$$p_1 = p_0 \frac{273,15 + \vartheta}{273,15} \ Amontons' Law \tag{1.17}$$

It is apparent for formal reasons that at $\vartheta = -273.15\,^\circ\text{C}$ also the volume and the pressure of the ideal gas disappear. From this it can be concluded that the temperature $\vartheta = -273.15\,^\circ\text{C}$ represents a physical limit for the temperature (absolute zero).

The laws of Charles and Amontons can be combined as

$$V = \frac{V_0 T}{T_0} \text{ (Charles)} \tag{1.18}$$

and

$$p = \frac{p_0 T}{T_0} \text{ (Amontons)} \tag{1.19}$$

One then obtains an equation of state which contains all state variables (p, V, T):

$$\frac{pV}{T} = \frac{p_0 V_0}{T} = \text{constant} \tag{1.20}$$

The Italian physicist Avogadro discovered in 1811, that the pressure of a gas is proportional to the number of molecules present:

$$\frac{p_1}{N_1} = \frac{p_2}{N_2} Avogadro's\ Law \ (T, V \text{ constant}) \tag{1.21}$$

At a 'standard temperature' of (0 °C) and a 'standard pressure' of (101 300 Pa) 1 mol of a gas always contains $N = 6.02252 \cdot 10^{23}$ particles taking up the volume of (V_{molar}) 22.4136 l (molar volume).

When inserting for p the 'standard pressure p_n', for T the 'standard temperature T_n,' and for V the 'molar volume V_{molar}', then one obtains the 'universal or molar gas constant R' for ideal gases:

$$R = \frac{p_n V_{molar}}{T_n} = \frac{101300\text{Pa } 22.414\text{ m}^3}{273.15\text{ K kmol}} = 8.314\frac{\text{kJ}}{\text{kmol K}} \tag{1.22}$$

or

$$R = 83.14\frac{\text{mbar L}}{\text{mol K}} \tag{1.23}$$

Since the molar volume can also be expressed as the volume of the gas divided by the number of moles (n):

$$V_{molar} = \frac{V}{n} \tag{1.24}$$

one obtains from Eqs. (1.20) and (1.22) the universal equation of state for ideal gases:

$$pV = nRT \tag{1.25}$$

This equation of state which in effect only applies to 'ideal gases' may in a vacuum be applied with good accuracy also to real gases and vapours, because in vacuum the interactions between the particles reduce owing to the increasing free path. For this reason, this equation of state can be used to calculate pumped gas, respectively vapour quantities as they occur in connection with chemical engineering processes.

1.2.3 Flow of Gases through Pipes in a Vacuum

The flow conditions within a gas can be described through the Knudsen number. Flows through pipes are characterised through the Reynolds number. At relatively high pressures in a rough vacuum, a viscous flow is present which may either be laminar or turbulent. For a viscous flow, a mutual influence between the flowing particles is typical. In the turbulent range, the molecules behave chaotically. The Knudsen number is defined as follows:

$$Kn = \frac{\lambda}{d} \tag{1.26}$$

A viscous gas flow is characterised through a Knudsen number of $Kn < 0.01$.

The pipe diameter d is here much bigger than the mean free path λ of the molecules, and the gas flow is characterised by constant collisions amongst the particles.

The boundary between a continuous flow and a turbulent flow can be characterised through the Reynolds number (Re):

$$Re = \frac{u\rho d}{\eta} \tag{1.27}$$

ρ	=	gas density in kg/m^3
η	=	viscosity the gas
u	=	flow velocity
d	=	pipe diameter.

The following applies to the flow through pipes:

When the non-dimensional Reynolds number attains values of over 2200, then one speaks of a turbulent flow, in the case of values below 1200 one then speaks of a laminar flow. In the range in between, either turbulent or laminar flow conditions can be present.

When the mean free path λ of the molecules is equal to the pipe diameter d or exceeds it, then there results a Knudsen number of >1 and when the Reynolds number is <1200 then the behavior of the gas molecules becomes more and more individual, that is, the flow changes to a 'molecular flow'.

1.2.3.1 Gas Throughput and Conductance

The term gas throughput is defined as a quantity of gas flowing through a given area per unit of time.

The gas quantity Q corresponds here to the volume ($\dot{V}$) of gas occurring per unit of time multiplied with the prevailing pressure p and Eq. (1.29):

$$Q = p\dot{V} \tag{1.28}$$

The SI unit for the gas throughput Q is Pa m^3 s^{-1}.

The gas throughput corresponds to an energy passing through an area per unit of time [3]

$$1\,\mathrm{Pa\,m^3\,s^{-1}} = 1\,\mathrm{W} \tag{1.29}$$

From Eq. (1.28) one then obtains the gas throughput as a mass flow:

$$\dot{m}\,\mathrm{kg\,s^{-1}} = \frac{\dot{Q}M_{\mathrm{molar}}}{RT} \tag{1.30}$$

M_{molar} = molar mass of the gas.

The transported gas quantity Q, which flows through a pipe is in the case of a continuous flow dependent on the prevailing pressure difference between inlet and outlet.

When taking the ratio between the gas quantity being throughput and the prevailing pressure difference across the pipe, then one obtains a quantity which can be designated as 'conductance' L of this pipe:

$$L = \frac{Q}{\Delta P} \tag{1.31}$$

Equation (1.31) is also termed 'Ohm's Law' of vacuum technology. When now considering the flow of a gas through an orifice (very short pipe) one obtains a rather complicated dependency of the gas being throughput as a function of the pressure

difference ahead of, and after the orifice. Let the pressure ahead of the orifice be the atmospheric standard pressure, whereas the pressure is being reduced behind the orifice. Through the reduction in 'flow out pressure' the gas quantity being put through will increase until it attains a maximum. One now speaks of a critical pressure ratio, the gas then flows through the orifice at the speed of sound.

The gas quantity now passing through the orifice can be calculated from [3]

$$Q = Ap_1C'\left(\frac{2\kappa kT}{(\kappa-1)\,m}\right)^{\frac{1}{2}}\left(\frac{p_2}{p_1}\right)^{\frac{1}{\kappa}}\left[1-\left(\frac{p_2}{p_1}\right)^{\frac{(\kappa-1)}{\kappa}}\right]^{\frac{1}{2}} \tag{1.32}$$

where $\kappa = C_p/C_V$, A = area of the orifice, p_1 = pressure ahead of the orifice, p_2 = pressure downstream of the orifice, C_p, C_V = heat capacity at constant pressure, respectively constant volume.

C' is a reduction factor since the gas which passes at a high velocity through the orifice, is subject to a volume contraction downstream of the orifice ('vena contracta').

For thin orifices, C' has a value of approximately 0.85.

When after attaining the speed of sound, the flow out pressure is reduced further, then the gas throughput will not increase further. Under these conditions the following applies

$$\frac{p_2}{p_1} = \left(\frac{2}{(\kappa+1)}\right)^{\frac{\kappa}{\kappa-1}}$$

and one speaks of the critical expansion ratio.

The gas flow Q which is then being put through becomes

$$Q = Ap_1C'\left(\frac{kT}{m}\frac{2\kappa}{\kappa+1}\right)^{\frac{1}{2}}\left(\frac{2}{\kappa+1}\right)^{\frac{1}{(\kappa-1)}} \tag{1.33}$$

And is thus independent of the flow out pressure p_2.

Equation (1.33) represents the critical gas flow. For air, $\kappa = 1.4$.

And blocking takes place at $p_2/p_1 = 0.525$. Figure 1.3 clarifies the flow under blocking conditions.

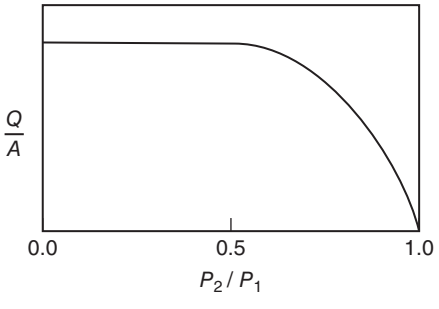

Figure 1.3 Gas flow under blocking conditions after attaining the critical pressure ratio.

These flow resistance considerations are important to vacuum systems when through these, gas flows are restricted in connection with pump-down processes or venting processes or when measuring leaks, for example.

1.2.3.2 Flow through Long, Round Pipes

The most well-known mathematical approach describing a viscous flow through long, straight pipes is through the so-called *Hagen–Poiseuille equation:*

$$Q = \frac{\pi d^4}{128\eta l}\frac{p_1 + p_2}{2}(p_1 - p_2) \tag{1.34}$$

The conductance L of a cylindrical pipe for air at 0 °C is stated in litres per second whereby diameter d and pipe length l are entered in centimetres and the pressure p is entered in millibars as follows [4]:

$$L = 135\frac{d^4}{l}\frac{p_1 + p_2}{2}$$

This special solution is valid, provided the following additional conditions are fulfilled:

- The flow profile in the pipe is fully present (location independent, applies to long pipes).
- The flow conditions are laminar, that is, $Re < 1200$ and $Kn < 0.01$
- The ratio between the actual gas velocity and speed of sound is <0.3.

A comprehensive description of the flow conditions in gas flows also through conducting sections exhibiting different geometries under different pressure and flow conditions can be found in [2, 3].

Analogously to 'Ohm's Law' the conductance changes when connecting pipes, valves or equipment like condensers as follows:

- Conductances add for a parallel connection:

$$L_{\text{tot}} = L_1 + L_2 + \cdots L_i \tag{1.35}$$

- Conductances add reciprocally for a series connection:

$$\frac{1}{L_{\text{tot}}} = \frac{1}{L_1} + \frac{1}{L_2} + \cdots \frac{1}{L_i} \tag{1.36}$$

Figure 1.4 states the conductances for pipes of commonly used nominal widths for a laminar flow [5].

If in the pipe, bends and components like valves are present then a correction is introduced by assuming a longer length of the pipe ('equivalent pipe lengths').

In Figure 1.5 the equivalent pipe lengths of valves and pipe bends are stated. As to the conductance values for equipment it will be necessary to ask the manufacturers for such information. As a rule, all vacuum lines should be as short as possible and have a diameter which is as large as possible (same dimension as the intake port on the vacuum pump used).

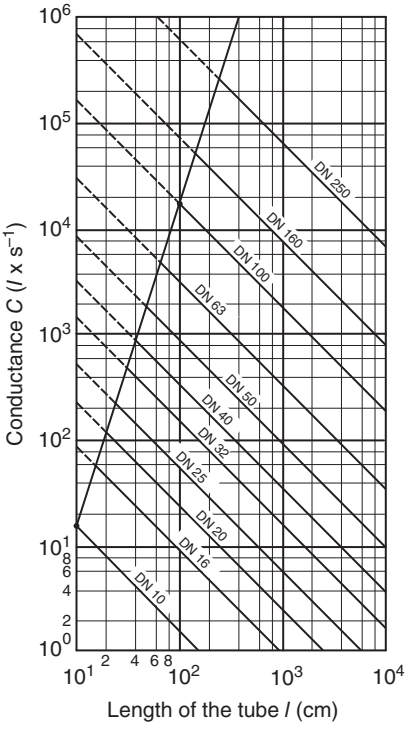

Figure 1.4 Conductance values of pipes of common nominal widths with a circular cross-section (apply to a laminar flow ($p = 1$ mbar), flowing medium is air).

DN	10	16	25	40	65	100	150	250
90° elbows; $D = 3\,d$	0.2	0.3	0.4	0.6	1.0	1.5	2.5	3.1
Straight-line valve	0.8	0.9	1.0	1.1	1.6	2.4	3.8	5.2
Right-angle valve	0.7	0.8	0.9	1.0	1.4	2.0	2.6	3.3

Figure 1.5 Equivalent pipe lengths in metres for components.

In the case of a molecular flow, the conductance becomes independent of the mean pressure difference across the length of the pipe [4]:

$$L = 12.1\frac{d^3}{l} \text{ in litres per second} \tag{1.37}$$

1.2.4 Vacuum Pumps Overview

The standard DIN 28400 Part 2 provides an overview of commonly used vacuum pump types.

However, in the area of chemical process engineering only pumps of the following general types are used:

- *Positive displacement pumps:* diaphragm vacuum pumps, liquid ring vacuum pumps, rotary vane and rotary piston vacuum pumps as well as Roots vacuum pumps (dry compressing claw and screw pumps are not yet mentioned here but also belong to this group of pumps).
- *Kinetic vacuum pumps:* vapour jet pumps, gas ejector pumps and liquid jet pumps.
- *Adsorption pumps:* the condenser.

The specific design types of different vacuum pumps, their special characteristics and application in the area of chemical process engineering are covered in the following chapters of this book.

The areas of application for these vacuum pumps are all within the rough and medium vacuum range, the principal vacuum range for chemistry processes. Only the short path and molecular distillation processes rely also high-vacuum pumps like the diffusion pump or even the turbomolecular pump (kinetic gas pumps). High-vacuum pumps are not covered in this book. For these refer to [2, 3].

It is the task of all vacuum pumps outlined above to generate a vacuum in a process engineering plant, for example, and to provide enough pumping speed. A pumping speed is in the end only a volume which passes per unit of time through an area.

Or the pumping speed S of a vacuum pump corresponds to the gas throughput Q, which passes through the area of the intake port of a vacuum pump, for example within a certain time ($\dot{Q}$), divided by the prevailing pressure.

$$S = \frac{\dot{Q}}{P} \tag{1.38}$$

The pumping speed S of a vacuum pump operating in the rough and medium vacuum range is commonly stated in m^3h^{-1}.

Figure 1.6 depicts the so-called pumping speed diagram of a mechanical rotary vane vacuum pump. The pumping speed is graphed as a function of the intake pressure.

The evacuation process for a vacuum chamber, like that of a distillation column, is effected with a rough vacuum pump, regardless of type, as follows:

The gas quantity $-(V_K \Delta p)/\Delta t$ pumped out per unit of time Δt from a constant distillation column volume V_K flows at the prevailing pressure in the distillation column into the intake port of the vacuum pump. The corresponding gas quantity Q is equal to the effective pumping speed S_{eff} of the vacuum pump, respectively the pump system connected to the vacuum chamber (due to low conductance values