VACUUM GUIDE

UNDERSTANDING THE BASICS

EDWARDS

EDWARDS
THE PARTNER OF CHOICE

Edwards, a world leader in vacuum solutions and innovator in vacuum pump design and manufacturing, has been delivering solutions that bring value to manufacturing industries for more than 100 years.

We believe in delivering results that bring value to our customers by using our breadth of industry experience to identify and apply solutions to your problems. Using the most innovative and up-to-date modelling techniques, we optimise the pumping configuration for customers to provide a system design giving the maximum performance in the most reliable and cost-effective way.

CONTENTS

1. INTRODUCTION

Vacuum is at the heart of research and development, in pioneering fields such as high energy physics and space simulation; and at grass roots level, where we foster experimentation in the laboratories of schools and universities. At Edwards we create vacuum equipment to meet these exacting demands, through off-the-shelf or bespoke solutions; from initial advice through to implementation and support, we deliver safe, stable vacuum environments that keep up with the complex and evolving demands on both analysis and discovery.

Equally, vacuum plays an important role in the chemical industry, *e.g.* for the manufacturing of active pharmaceutical ingredients (APIs), freeze drying, and central vacuum systems. It is used in a variety of chemical processes: upstream to assist the synthesis of chemicals by removal of air/oxygen, material transfer or reactor loading, and downstream during separation and purification of the target product through distillation, filtration or drying, as well as for raw material recycling.

With more than 100 years of expertise in developing vacuum pumps, our experience safeguards a reliable and stable vacuum environment. To us, partnership-like customer relationships are fundamental. Therefore, this vacuum guide is meant to provide an overview of vacuum technology in an easy to read manner.
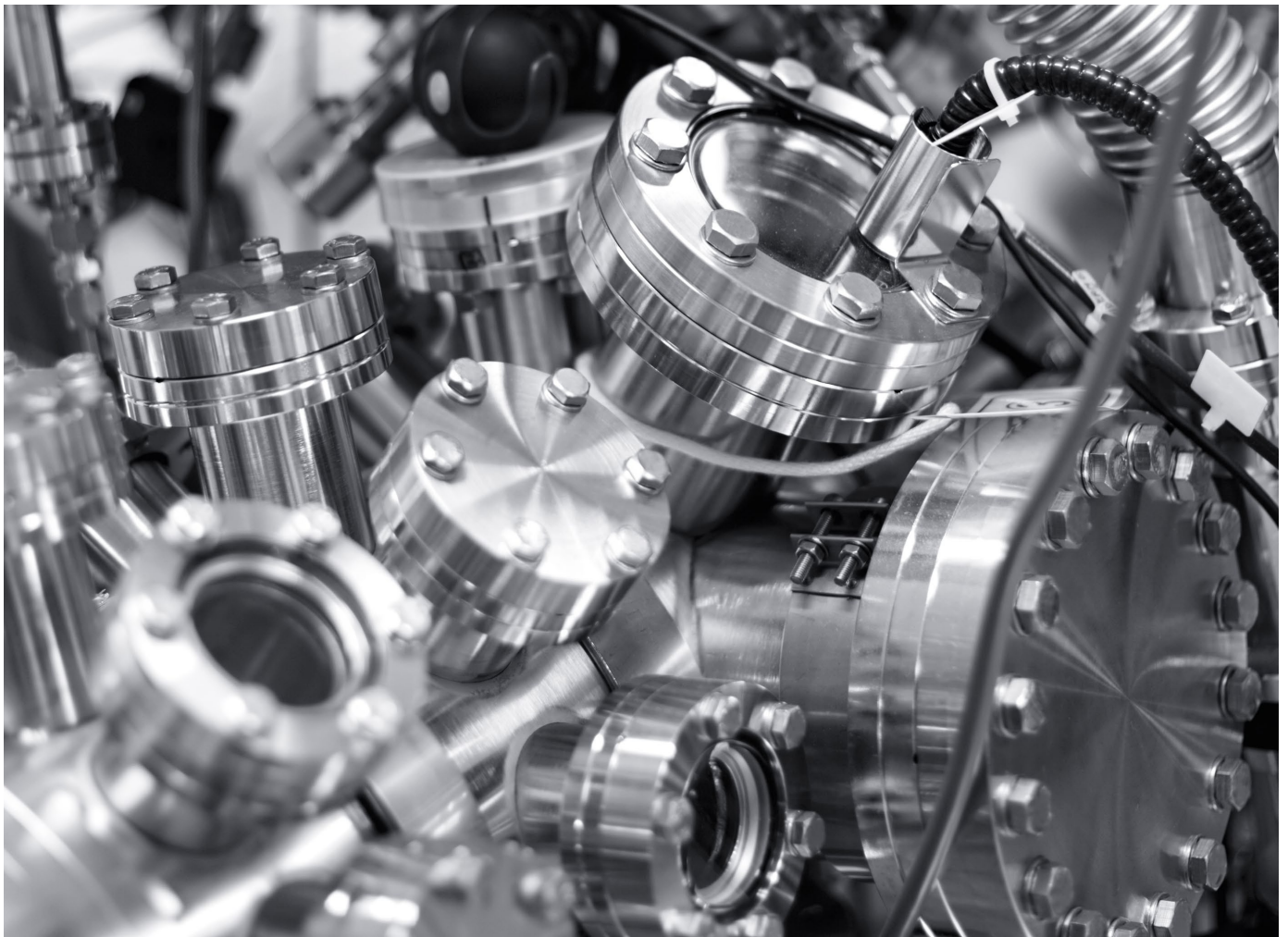
2. FOUNDATIONS OF VACUUM TECHNOLOGY

2.1 DEFINITION OF VACUUM

Let us start by giving you some perspective. A practical vacuum is achievable from 10^3 to $< 10^{-12}$ mbar(a) – a dynamic range of no less than 15 orders! There are few disciplines of science that fall within such a wide working range.

The selection process for pumps therefore necessitates a familiarity, appreciation and understanding of the basic principles and terminology of vacuum science which is what will be discussed next.

A vacuum is described as a space in which the pressure is below the surrounding atmospheric pressure. Within a gas the total pressure is defined as the rate of transfer of the normal component of momentum of the constituting molecules to a plane per unit area. The SI unit of pressure being the Pascal (Pa), which is the pressure exerted by a force of one Newton perpendicular to a plane surface of 1 m².

By definition:

1 standard atmosphere $= 1.01325 \times 10^5$ Pa
$= 1013.25$ mbar(a)
$= 760$ Torr

N.B. increasingly the unit hPa is used where 1 hPa = 1 mbar(a)

A common scheme of the vacuum range is shown in Table 2.1:

Low or Rough Vacuum (LV)	Atmospheric to 1 mbar(a)
Medium or Fine Vacuum (MV)	1 to 10^{-3} mbar(a)
High Vacuum (HV)	10^{-3} to 10^{-7} mbar(a)
Ultra High Vacuum (UHV)	10^{-7} to 10^{-12} mbar(a)
Extreme High Vacuum (XHV)	$< 10^{-12}$ mbar(a)

Table 2.1: Definition of vacuum ranges.

Orbit of meteo satellites

36,000 km

Ultra High Vacuum <10^{-10} mbar

Orbit of satellites
close to Earth

200 to 1000 km

High Vacuum <10^{-4} mbar

Orbit of International
Space Station

400 km

High Vacuum <10^{-4} mbar

Altitude of Stratos Balloon

31 km

Medium Vacuum <10 mbar

Usual cruising altitude of
passenger airplanes

8 to 12 km

Rough Vacuum ~ 290 mbar

Highest peak in the Alps
(Mont Blanc)

4.81 km

Rough Vacuum ~ 560 mbar

Sea level

0 km

1013 mbar (norm value)

Ultra High Vacuum

High Vacuum

Medium Vacuum

Rough Vacuum

10^{-12}
10^{-10}
10^{-8}
10^{-6}
10^{-4}
10^{-2}
1
10
50
100
250
500
1.000
mbar
hPa

2.2 CONCEPTS & TERMINOLOGY

In this section we discuss some important vacuum concepts and illustrate how they impact the mechanism, choice and use of vacuum pumps.

The vacuum environment is essentially a gas at a reduced pressure. Theories of vacuum must therefore account for the laws which have been shown to hold true for gases. To describe the properties of a gas, the equation of state for ideal gases is commonly used. It relates the pressure, p, volume, V, and temperature, T, of a gas. One form of the equation of state of ideal gases is:

$$p = nk_0T \qquad (2\text{-}1)$$

where:

p = the pressure,

n = the number density,

k_0 = the Boltzmann's constant, and

T = the absolute thermodynamic temperature

The number density, n, is the number of molecules, N, per unit volume, V:

$$n = \frac{N}{V} \qquad (2\text{-}2)$$

and $n = 2.5 \times 10^{16} \times p$ per cm^{-3} at 20°C where p is in mbar(a) as illustrated in Table 2.2:

Pressure (mbar(a))	Number density (molecules/cm^{-3})
10^3	2.5×10^{19}
1	2.5×10^{16}
10^{-6}	2.5×10^{10}
10^{-10}	2.5×10^{6}

Table 2.2: Pressure with according number density.

Depending on the physical parameters, the behaviour of real gases can deviate from that of an ideal gas. For example, with air under ambient conditions, the value of the product pV is about 0.02% smaller than assumed for an ideal gas. At higher pressures and lower temperatures, deviations from ideal gas behaviour become larger. Since vacuum technology deals with diluted gases at ambient or elevated temperatures, in which the particle density is small and the mean thermal velocity is high, the intrinsic volume and intrinsic pressure (caused by van-der-Waals force) of the gas particles can be neglected.

Aside - a more convenient expression of equation (2-1) is shown below, derived using $k_o = R_o/N_A$ where n_m is the number of moles of the gas ($n_m = N/N_A$), R_o is the gas constant and N_A is Avogadro's number:

$$pV = n_mR_0T \qquad (2\text{-}3)$$

It can be seen that the product pV is proportional to the number of moles present and equals the 'quantity', Q, of the gas. This important concept will be discussed further below. Even at low pressures there are still a very large number of entities (molecules) allowing significant statistical analysis.

Kinetic Theory gives several relevant outcomes for vacuum science:

i. The mean thermal velocity $\bar{v}$ is given by

$$\bar{v} = \left(\frac{8R_0T}{\pi M}\right)^{1/2} \qquad (2\text{-}4)$$

where M is the molar mass of the gas.

For example, at 20°C, $\bar{v}$ for a helium molecule is 1,245 m/s compared to 470 m/s for nitrogen and 394 m/s for argon.

ii. We can view gases as moving at random with relatively high velocities and collisions occurring between them as shown below in Fig. 2.1.
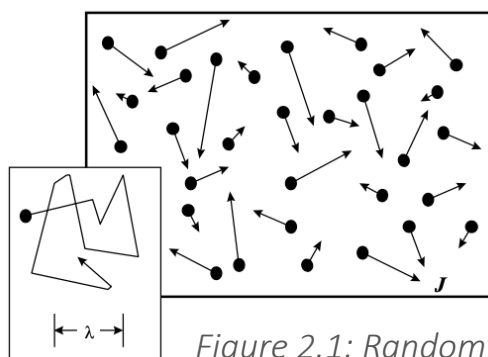

Figure 2.1: Random movement of molecules.

This allows us to consider the concept of mean free path which is defined as the mean distance travelled by molecules between collisions:

$$\lambda = \frac{k_0T}{\sqrt{2}\pi p d_m^2} \qquad (2\text{-}5)$$

where:
λ = the mean free path, and
d_m = the molecular diameter.

Additionally, we can define the impingement rate, J, as the number of molecules intersecting or impinging on a vacuum surface where:

$$J = 0.25 \cdot n\bar{v} \qquad (2\text{-}6)$$

Extending this analysis we develop the monolayer formation time, t_{mono}, which is the time taken for the formation of one (uniform) adsorbed layer of gas on a vacuum surface.

These parameters are summarised in Table 2.3:

Pressure (mbar(a))	Impingement rate (cm^{-2}.s^{-1})	Mean free path	Monolayer formation time
10^3	3×10^{19}	66 nm	3 ns
1	3×10^{16}	0.066 mm	3 ms
10^{-3}	3×10^{13}	6.6 cm	3 ms
10^{-6}	3×10^{10}	66 m	3 s
10^{-10}	3×10^6	660 km	8 hours

Table 2.3: Parameters for the monolayer formation time (for N$_2$ at 20°C).

iii. Flowing gas

We define the flowrate or throughput, Q, of a gas as the quantity/unit time (in mbarm³/h, kg/h or slm):

$$Q = pV = pS \qquad \text{(2-7)}$$

where speed, S = volume rate and the conductance, C, is a measure of the ease of gas flow between two regions with a pressure difference of Δp of such that:

$$C = \frac{Q}{\Delta p} \qquad \text{(2-8)}$$

The effective or net pumping speed (volumetric flowrate), S_{net}, can be significantly affected by the conductance of connecting components.

Conductance   **Pump**

C S
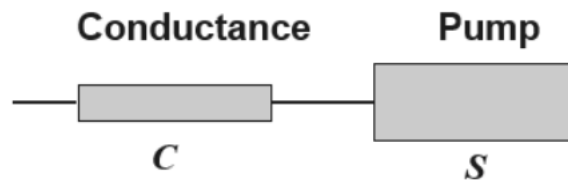
Pumping speed can be combined with a conductance in the same way as conductances in series.

$$\frac{1}{S_{net}} = \frac{1}{S} + \frac{1}{C} \qquad \text{(2-9)}$$

Fig. 2.2 shows the restrictive effect of a 1 m length NW40 connecting pipe on the net pumping speed ($S_{upstream}$) of various speed turbomolecular pumps ($S_{downstream}$):
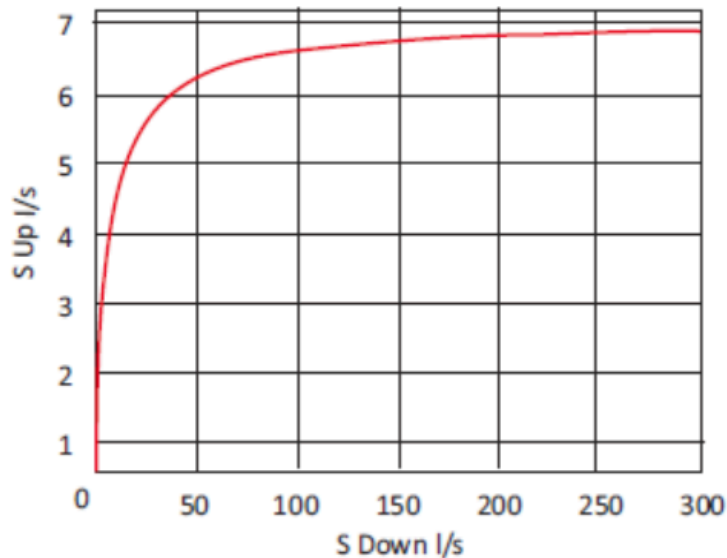

Figure 2.2: Restrictive effect of a 1 m length NW40 pipe on the net pumping speed.

iv. Flow regimes

We have developed a picture of gas molecules moving randomly at high velocities with a wide range of defining parameter values, across the vacuum spectrum. To deliberately induce flow, *i.e.* to create a pumping effect, we need to exploit and influence these.

Reconsidering the concept of mean free path we can see that for a typical vacuum component with a dimension, d, of 7 cm (*e.g.* the diameter of an ISO63 pipe) molecules will be more likely to collide with other molecules than with the pipe wall, but decreasingly so as the pressure reduces to 10^{-3} mbar(a).

- We introduce the concept of the Knudsen number, K_n:

$$K_n = \frac{\lambda}{d} \qquad (2\text{-}10)$$

- As shown in Fig. 2.3, as vacuum improves (and system pressure decreases), collisions within the volume become relatively few compared with surface collisions.
- The ratio of surface to volume collisions can be shown for a box to be ~ 3 x K_n allowing us to define flow regimes as below:

$$K_n \gg l, \lambda \gg d \qquad K_n \ll l, \lambda \ll d$$

molecule-surface   molecule-molecule

collisions dominate   collisions dominate

Molecular   **Continuum**

Flow regime

Molecular   Transitional   Continuum

Increasing pressure

Typically < 10^{-3} mbar(a)   Typically > 1 mbar(a)
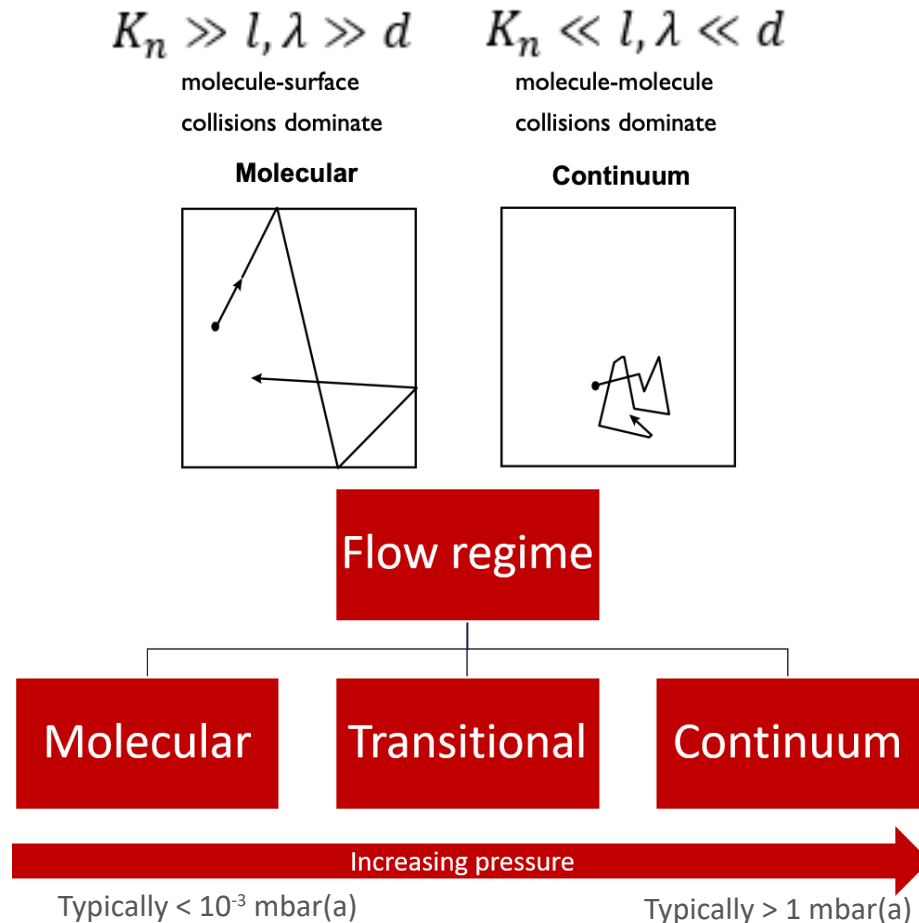
Figure 2.3: Different flow regimes dependent on the pressure.

The understanding of flow types is a fundamental requirement in the design of vacuum pumps and systems, both in terms of the different mechanisms employed to create controlled gas flow at different pressures and also the optimisation of system design.

v. Compression ratios

The pumping process relies on the compression of gas. The pressure ratio of a pump mechanism's exhaust pressure and the inlet pressure is known as the compression ratio, $K = P_{exhaust}/P_{inlet}$. The maximum compression ratio is referred to as K_o and is the compression with no gas flow at the inlet. The theoretical definition of compression for a given pump or pumping stage depends on the type of mechanism producing the compression; the compression ratio or a pump mechanism may be different for different gases. It is important to consider the effect and mitigation of the internal heat of compression within any compressing mechanism.

2.3 CLASSIFICATION OF PUMP TECHNOLOGIES

A consequence of the kinetic nature of gases is that there are different flow types across the vacuum spectrum, and these are exploited accordingly in the design of suitable pumping mechanisms for given vacuum and process conditions. The diagram shown in Fig. 2.4 is a schematic representing the most common pump classifications and families.

Fig. 2.4: Most common types of vacuum pumps.
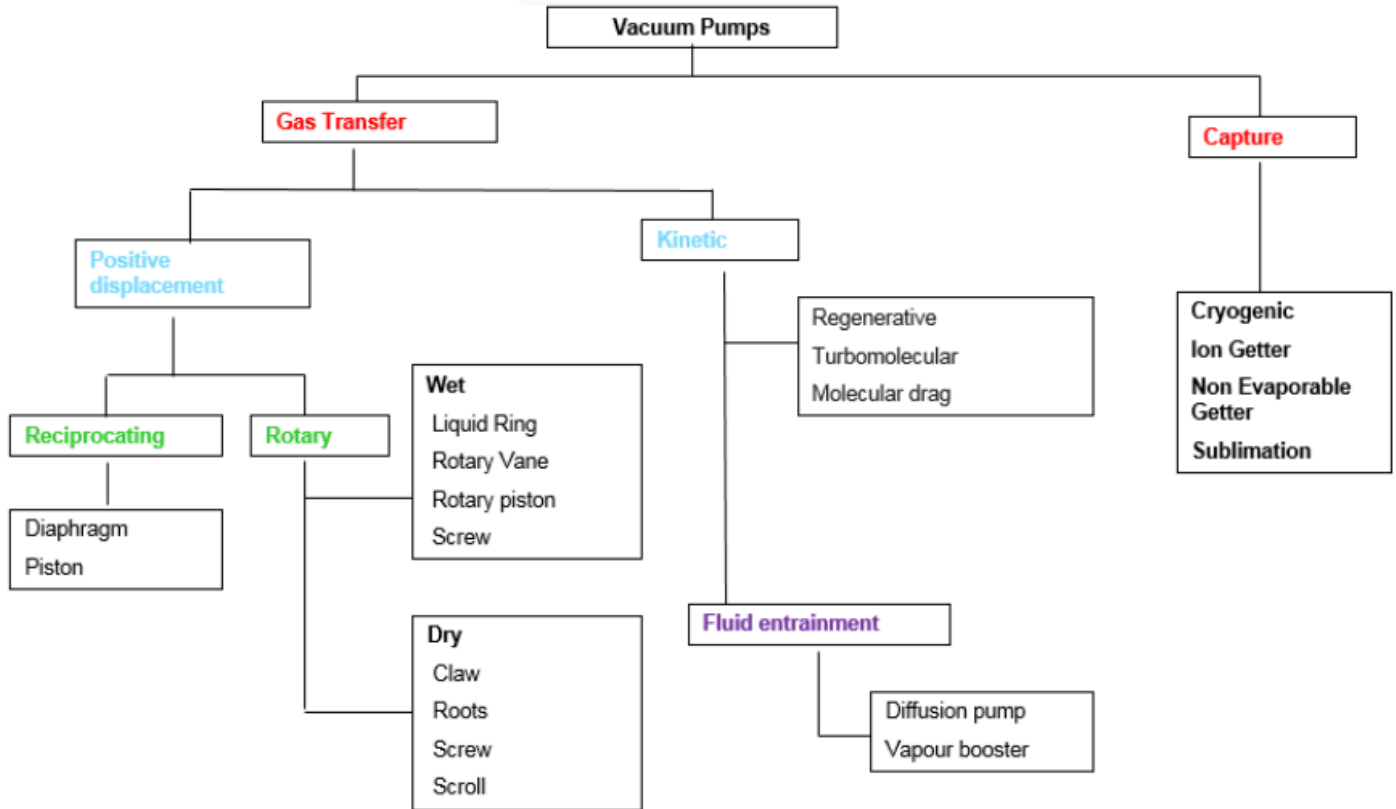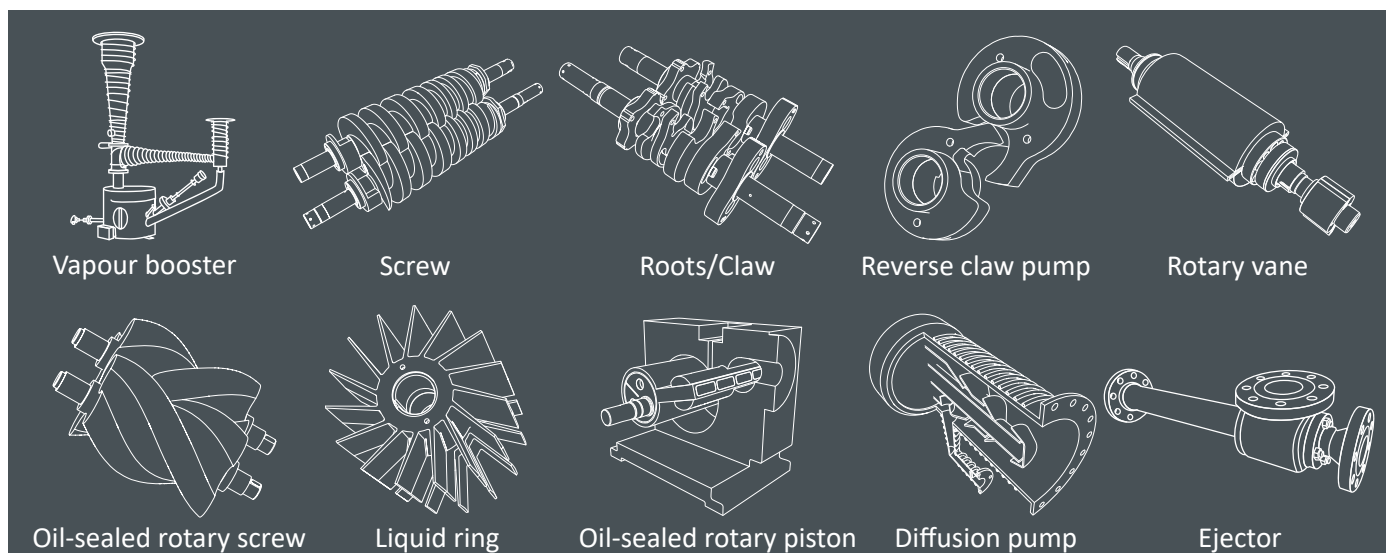
Typically, the different pressure ranges in vacuum technology are defined as follows: rough vacuum (from 10^3 mbar(a) to 1 mbar(a)); medium vacuum (1 mbar(a) to 10^{-3} mbar(a)); high vacuum (10^{-3} mbar(a) to 10^{-7} mbar(a)); ultra-high vacuum (10^{-7} mbar(a) to 10^{-12}); and extreme high vacuum (less than 10^{-12} mbar(a)) *(see Table 2.4 with working ranges)*.

	Ultrahigh vacuum $<10^{-7}$ mbar(a)	High vacuum 10^{-7} to 10^{-3} mbar(a)	Medium vacuum 10^{-3} to 1 mbar(a)	Rough vacuum 1 to 10^3 mbar(a)

Diaphragm pump
Liquid ring pump
Liquid jet pump
Rotary plunger pump
Piston pump
Scroll pump
Screw pump
Rotary Vane pump
Rotary Piston pump
Roots pump
Adsorption pump
Vapour jet pump
Diffusion ejector pump
Diffusion pump
Turbo molecular pump
Ion sputter pump
Cryo pump

10^{-13} 10^{-12} 10^{-11} 10^{-10} 10^{-9} 10^{-8} 10^{-7} 10^{-6} 10^{-5} 10^{-4} 10^{-3} 10^{-2} 10^{-1} 10^0 10^1 10^2 10^3

Special operating range

pressure p [mbar(a)]

Standard operating range

Table 2.4: Working ranges of different pump technologies.

Besides the vacuum pressure ranges that can be obtained, it is also important to consider the performance envelope of different pump technologies. Depending on the size of the vacuum system, the economic perspective will further reduce the selection of applicable pump technologies.

Primary vacuum pumps (mainly working in the rough and medium vacuum range) are exhausting to atmospheric pressures and thus can act in isolation. Secondary pumps (typically working in the high and ultra-high vacuum range) continuously exhaust to a primary pump or require a primary pump to create a level of vacuum they can operate from.
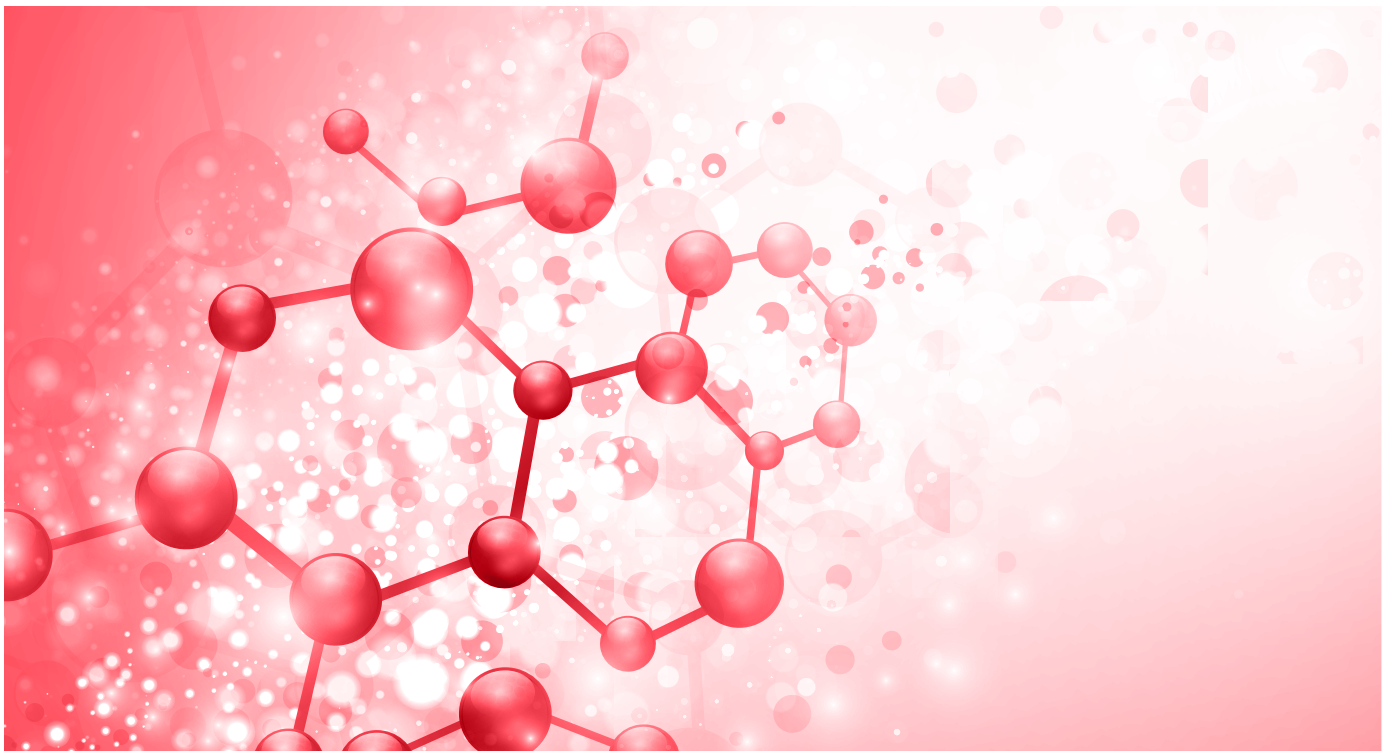
3. VACUUM PUMP TECHNOLOGIES

3.1 LIQUID SEALED ROTARY DISPLACEMENT PUMPS
3.1.1 LIQUID RING PUMPS

Liquid ring pumps (LRPs) usually use water as a sealing liquid. However, depending on the application other liquids, like oils or solvents, can be used. The pump features an eccentrically mounted impeller, which creates a pumping force as the liquid ring rises and falls. Fig. 3.1 depicts a schematic of a liquid ring pump.

The main advantage of LRPs is their capability of handling large amounts of vapour at high temperatures. They operate at low noise and vibration levels and can be built in a variety of different materials, making them suitable for a wide range of chemicals. Traditional once through LRPs require large amounts of sealing liquid. The fluid gets contaminated with process materials, which requires difficult and/or costly disposal. Alternatively, partial- or total-recirculation systems reduce the amount of consumed liquid.

LRPs are available with pumping capacities up to 50,000 m³/h with a pressure range down to 33 mbar(a). They are usually in a single or double-stage set-up and may be combined with dry mechanical booster pumps or steam ejectors for low pressure operation.
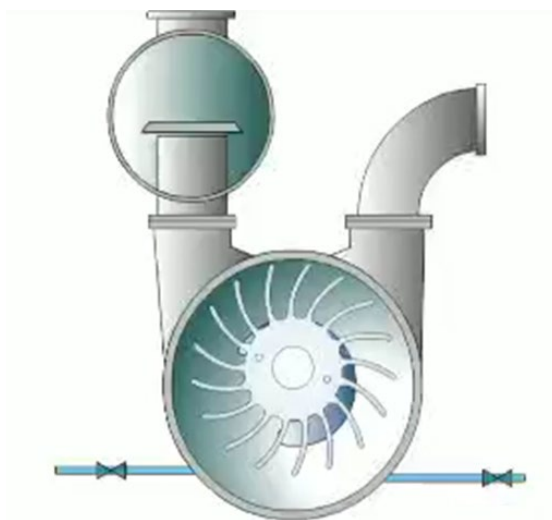
Figure 3.1: Schematic of a liquid ring pump, featuring an eccentrically mounted impeller.

Application area	• Petrochemical, chemical, pharmaceutical & fine chemical applications • Power generation • Offshore applications • Food processing
Pressure range	10^3 to 33 mbar(a)
Disadvantages	Advantages
• Limited ultimate pressure • High effluent generation • Sensitive to changes in effluent temperature	• Large capacity range • Wide range of construction materials • Very tolerant to process upsets (liquid & solid carryover) • Low noise & vibration

3.1.2 ROTARY VANE PUMPS

Rotary vane pumps are the most common type of positive displacement vacuum pumps. As a very mature technology, they offer excellent reliability, robustness, compact design and low costs of investment.

Operating within a pressure range from atmosphere down to approx. 10^{-4} mbar(a), makes them ideal backing pumps for any kind of medium and high vacuum pumps.

Fig. 3.2 shows the operating principle of a rotary vane pump. As the rotor with the vanes rotates, the gas is moved from the inlet and compressed towards the outlet.

Whereas the oil-sealed operation is a disadvantage for some applications, the use of oil enables higher compression ratios, better internal cooling behaviour and makes the pump resistant to dirt, dust and condensate.

Of course, the need for servicing the pumps regularly (oil changes) means higher costs of ownership – compared to dry pumps of similar size.
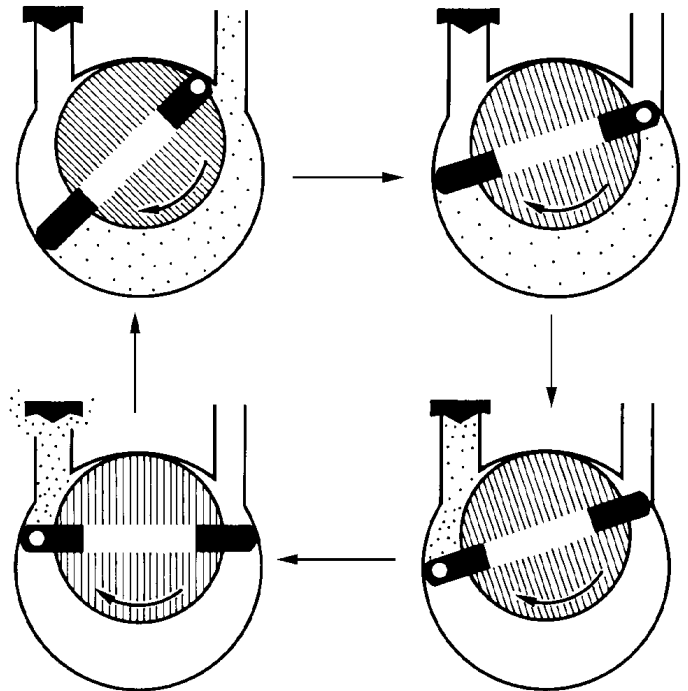
Figure 3.2: Schematic of a rotary vane pump's operating principle.

Application area	• Industrial & coating applications • Analytical instruments (mass spectrometers, leak detectors,…) • Research & development • Semiconductor and solar applications (special versions)…	
Pressure range	10^3 to 10^{-4} mbar(a)	
Disadvantages	**Advantages**	
• No contamination-free vacuum (hydrocarbons) • High cost of ownership (oil changes, maintenance)	• Very low ultimate pressure • High pumping speed (from atm) • Low power consumption • Low capital costs	

3.2 DRY COMPRESSING ROTARY DISPLACEMENT PUMPS

3.2.1 CLAW PUMPS

Claw pumps operate with one or multiple sets of non-contacting claw rotors. The mechanisms inlet and exhaust are arranged axially. As the two rotors rotate in opposite directions, gas is drawn in through an inlet slot aligned with a cavity on one of the rotors. Further rotation closes the inlet while the claws compress the trapped volume of gas until the cavity in the second rotor exposes the outlet exhaust slot.

For compression from atmospheric down to less than one mbar(a), an all claw design requires three stages, which can be accommodated in one machine (see Fig. 3.3). The immediate and obvious benefit of the claw rotor design lies in the fact that by its nature it provides an inbuilt valve mechanism and thus is well suited to perform against high pressure differentials directly to atmosphere.

The reverse claw mechanism (an Edwards patented feature), operating with three stages on one shaft, with the second set of claws reversed, allows a direct gas path through the pump. A short, simple, high conductance gas path through the pump contributes to good vacuum performance and improved power utilisation. It eliminates possible corrosion sites and enables particles to pass easily through the pump. It also minimizes the area available for the build up of corrosives or condensable residue.
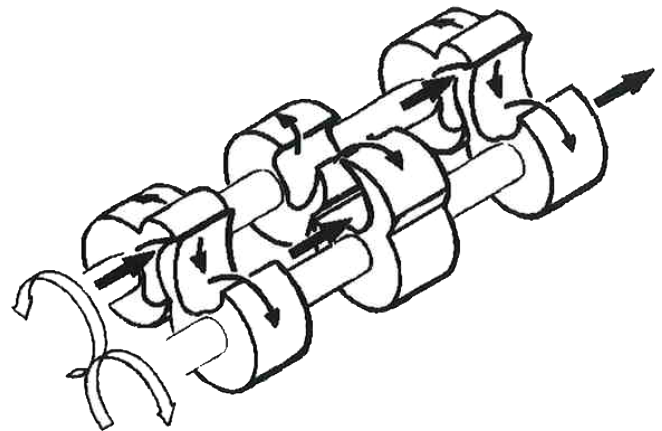
Figure 3.3: Three-stage claw pump with reversed claw stage.

Application area	• Petrochemical & chemical process industry • Pharmaceutical industry • Food processing • Plastic extrusion • Drying applications
Pressure range	Single-stage claw: 10^3 to 50 mbar(a) Multi-stage claw: 10^3 to 0.5 mbar(a)
Disadvantages	Advantages
• High capital costs • Limited capacity range	• Low cost of ownership / easy maintenance • Process gases can be maintained in the vapour phase (no corrosion, easy exhaust vapour recovery) • Environmental friendly (no effluent generation) • Safe pumping of flammable/corrosive mixtures without exotic materials

3.2.2 SCREW PUMPS

Screw pumps operate using two counter-rotating screw rotors (see Fig. 3.4), they are engineered so that as they rotate "in towards each other" they trap the gas in the volume between the "screws". This void decreases as the screws rotate which not only compresses the gas but moves it towards the exit port. They are frequently used as primary pumps for roots pumps.

Screw pumps have numerous important facets: Despite the micro-space between the two rotating screws, there are no contact parts nor is there any need for lubrication. As a result there is no contamination of the medium being pumped. Furthermore, rotary wear is minimal, they have a high tolerance against particles, employ high pumping speeds and achieve low ultimate pressures. They are highly efficient due to internal compression. However, they are less suitable for pumping light gasses, and cannot be easily scaled-down to lower pumping speeds. Operational costs and maintenance requirements are also relatively low.

Screw pumps are well suited for a wide range of industrial applications, which produce dust or where considerable quantities of condensate are generated.

Figure 3.4: Screw pump rotors.

Application area	• Industrial furnaces • Metallurgical systems • Packaging • Coating • Pharmaceutical & chemical processes
Pressure range	10^3 to 10^{-2} mbar(a)
Disadvantages	Advantages
• Limited capacity range • Poor capability to pump light gases	• Low operating costs • Process gases can be maintained in the vapour phase (no corrosion, easy exhaust vapour recovery) • Environmental friendly (no effluent generation) • Safe pumping of flammable/corrosive mixtures

3.2.3 ROOTS BOOSTER PUMPS

Roots blowers are commonly employed as 'booster' pumps on top of several types of primary pumps (*e.g.* rotary vane, screw and liquid ring pumps) to improve ultimate pressure and pumping speeds. Roots pumps employ two counter-rotating, interconnecting, lobes rotating within a chamber. Gas enters through the intake flange and is "trapped" between the two rapidly rotating units and the chamber wall, and is then expelled through (see Fig. 3.5).

1. Intake flange

2. Rotors

3. Chamber

4. Exhaust flange

5. Casing
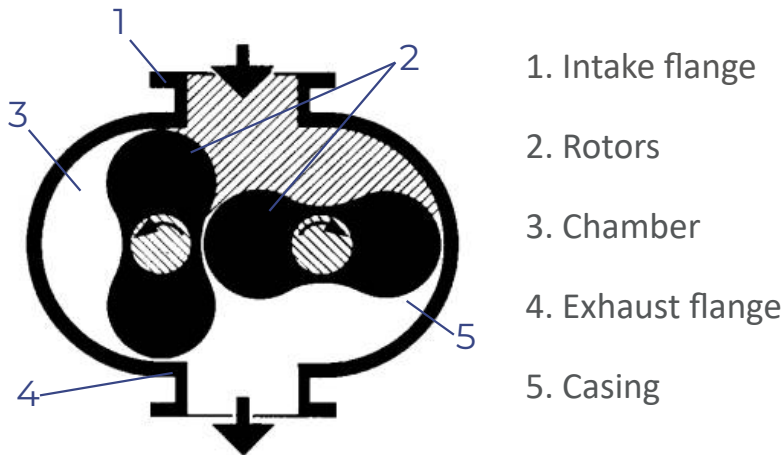
Figure 3.5: Roots booster working principle.

The advantages of roots pumps are that they are very quiet and compact, enjoy a long service life, there are no contacting parts, and they provide clean pumping (*i.e.* there are no particles or oils to contaminate the vacuum system). A roots-primary pump combination is typically a more economical option for improved pumping speeds and ultimate pressures compared to a bigger sized discrete primary pump. However, the limited compression ability means that a roots booster pump always needs a primary pump for operation.

Application area	• Industrial applications (laser industry, furnaces, metallurgy,…) • Pump systems with high pumping speeds (R&D & space, semiconductor, solar, central vacuum systems,…) • Processes with large quantities of gas/vapours	
Pressure range	10 to <10^{-4} mbar(a)	
Disadvantages	**Advantages**	
• (Always) needs a primary vacuum pump • Only small pressure differences are possible	• High volumetric capacities at low pressures • Multi-stage combinations with wet or dry backing pumps • Vapours kept in vapour phase (no exotic materials of construction required)	

3.3 FLUID-ENTRAPMENT PUMPS
3.3.1 DIFFUSION PUMPS

Diffusion pumps use a high-speed jet of vapour to direct gas molecules from the pump throat towards the bottom of the pump and out of the exhaust.

They have high pumping speeds and low costs when compared with other pumps in this vacuum range. However, because diffusion pumps cannot discharge directly into the atmosphere, a primary pump is required.

Fig. 3.6 shows the schematic of a diffusion pump. It operates with an oil of low vapour pressure, usually silicone oil or polyphenyl ethers. A high-speed jet is generated by boiling this oil and directing the vapour through a jet nozzle, where the gaseous flow changes from laminar and subsonic to supersonic-and-molecular, with several jets frequently being employed in series. The outside of the diffusion pump is cooled using either air-flow or a water jacket. As the vapour jet hits the outer cooled chamber of the pump, the vapour condenses and is recovered before being directed back to the boiler.

Diffusion pumps have no moving parts and as a result are durable and reliable. However, a major disadvantage of diffusion pumps is a tendency to backstream oil into the vacuum chamber resulting in carbonaceous or siliceous deposits.

Molecules of gas to be pumped
HV flange
Vapour jet
Cooling loops
Nozzle assembly
Backing pump connection
Pump body
Boiler
Heater
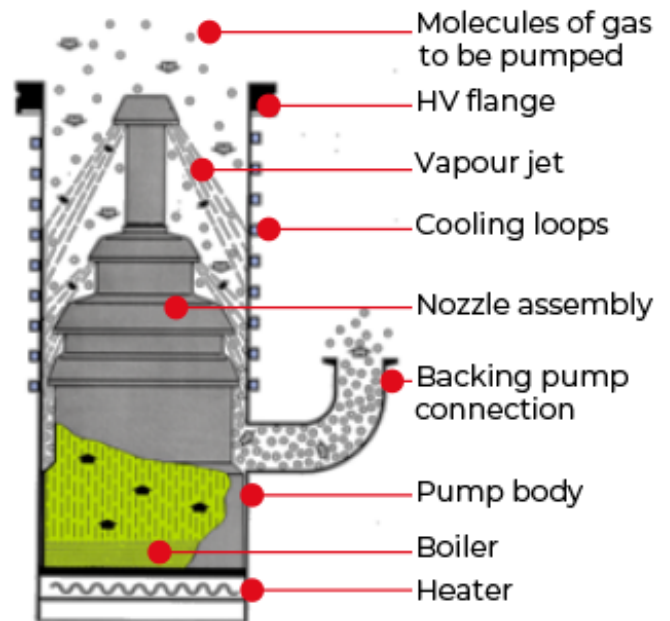
Figure 3.6: Operating system of oil diffusion pumps

Application area	• Metallurgy • Furnaces • Vacuum melting / drying • Vacuum coating • Space simulation	
Pressure range	$\sim 10^{-2}$ to 10^{-10} mbar(a)	
Disadvantages	**Advantages**	
• No hydrocarbon free operation, oil backstreaming • Requires backing pump • Heavy weight • Very sensitive to shock venting	• No vibration • Relatively low cost • Pumping all gases • Insensitive to heat, dust • High tolerance against radiation, magnetic fields	

3.3.2 STEAM EJECTORS

Steam ejectors basically consist of three parts: a nozzle, a suction chamber and a mixing diffuser (See Fig. 3.7). A high-pressure motive fluid, usually steam, is passed through the nozzle. The low-pressure vapour from the suction chamber is entrained in the steam and discharged at an intermediate pressure to another ejector (multi-stage system), to a condenser or to the atmosphere. Steam ejectors are highly reliable and robust, especially in demanding or corrosive environments. They provide rough to medium vacuum with large suction capacities. For the manufacturing of active pharmaceutical ingredients (APIs), a single-stage ejector cannot provide a low enough pressure. Therefore, multi-stage ejectors with direct or indirect interstage condensation need to be used.

Along with the high effluent generation, another disadvantage of steam ejectors is their high sensitivity to variations in process conditions and pressure. Their low thermal efficiency and high demand of steam and cooling water make them expensive to operate, especially in the case of multi-stage systems.

1	Strainer	5	Diffuser
2	Motive steam	6	Suction
3	Nozzle	7	Vapor Head
4	Discharge	8	Nozzle Holder
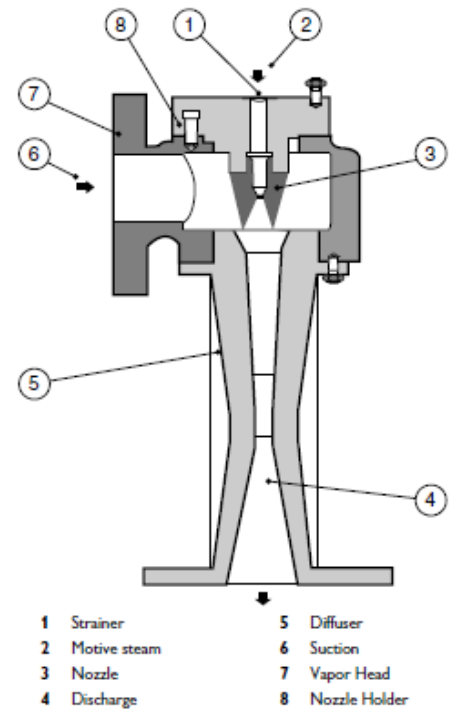
Figure 3.7: Schematic of a steam ejector

Application area	• Petrochemical & chemical process industry • Pharmaceutical applications • Power generation • Food processing • Seawater deaeration	
Pressure range	Single stage: 150-130 mbar(a) Two stage: 25-15 mbar(a) / Three stage: 10-7 mbar(a) Four stage: 1 mbar(a) / Five stage: 0.1 mbar(a)	
Disadvantages	**Advantages**	
• High effluent generation • High operating costs • Sensitive to changes in pressure & process conditions	• Large capacities and low pressures available with multi-stage combinations • Reliable, compact and simple to operate (no moving parts) • Available in a wide variety of materials	

4. VACUUM MEASUREMENT

The measurement of vacuum pressure plays an extensive and important role in all vacuum applications including for quality and safety.

When measuring a vacuum pressure, it must be remembered that there is no universal vacuum gauge – one that will respond accurately throughout the range from atmospheric pressure to 10^{-12} mbar(a). Therefore, understanding the application and the pressure measurement required is crucial to making the correct choices. Important considerations are the materials of construction, method of measurement (both direct and indirect), and electronics; all contributing to the limitation of each type of gauge.

Additionally, the environment which the gauge is situated can dictate the type suited *e.g.* radiation, magnetic fields, temperature and corrosive atmospheres.

4.1 SELECTION CRITERIA

The main criteria to consider when selecting a vacuum gauge are:

1. Pressure range: Ask yourself what the desired pressure range is that you want to measure, as different pressure ranges require the use of different gauges. Also consider the allowed measurement uncertainty / requested accuracy.

2. Application: Consider the specific application and process conditions you are using the vacuum gauge for. This includes amongst others:

> **a. Special operating conditions:** dirt/dust or vapours, vibrations and/or mechanical impact, strong gas flows, radiation, magnetic fields etc. have to be taken into account as some gauges are more sensitive towards environmental conditions than others.
> **b. Temperature:** Some gauges are more sensitive to temperature variations than others – so make sure the chosen gauge can withstand either hot or cold conditions if required, and the effect on accuracy which a change in temperature can give.
> **c. Gases:** Finally, if any special gases are involved in the system this also needs to be considered. The right correction factors must be used and materials selected to avoid any unwanted chemical reactions or media compatibility issues.

3. Communication & Control: This covers questions around how to control the measurements (*e.g.* controller type, cable length), the type of interface needed (*e.g.* RS232, Profibus, Devicenet,...), whether trigger points (set points, switching points) shall be used and so on.

4. Size, cost, serviceability

4.2 DIRECT OR INDIRECT

"Direct" gauges measure the force associated with pressure independently of the composition of the gas being measured. "Indirect" gauges are dependent on the gas species being measured, since they measure a pressure dependent property such as the thermal conductivity or ionization characteristics. Manufacturers offer combination gauges to overcome the measurement range limitations of some of the technologies. For instance, a combination Pirani-Penning gauge can operate from atmosphere to 10^{-10} mbar(a). Fig 4.1 gives an overview on the types of gauges.
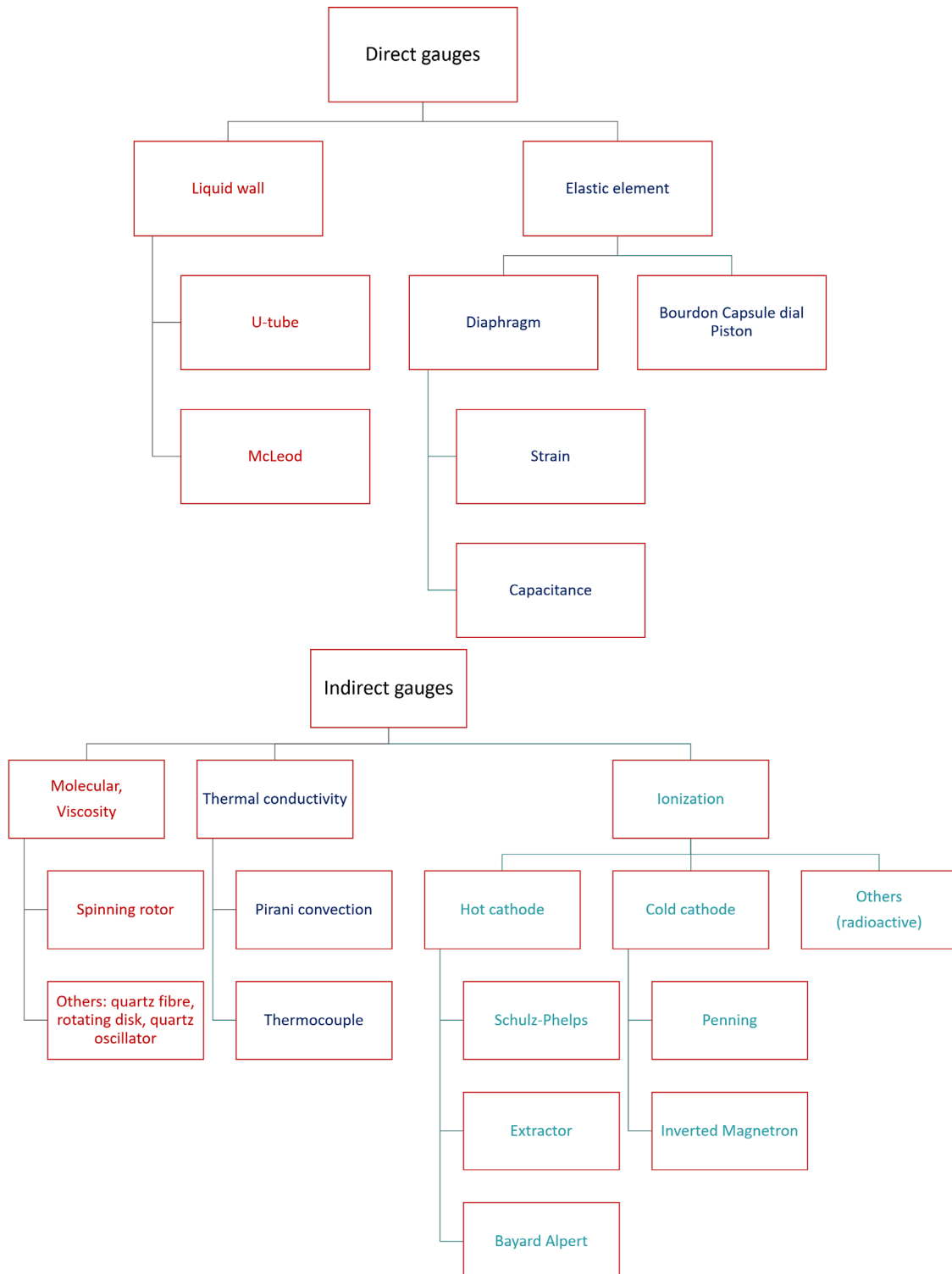

Figure 4.1: Classification of vacuum gauges.

4.3 MEASUREMENT RANGES

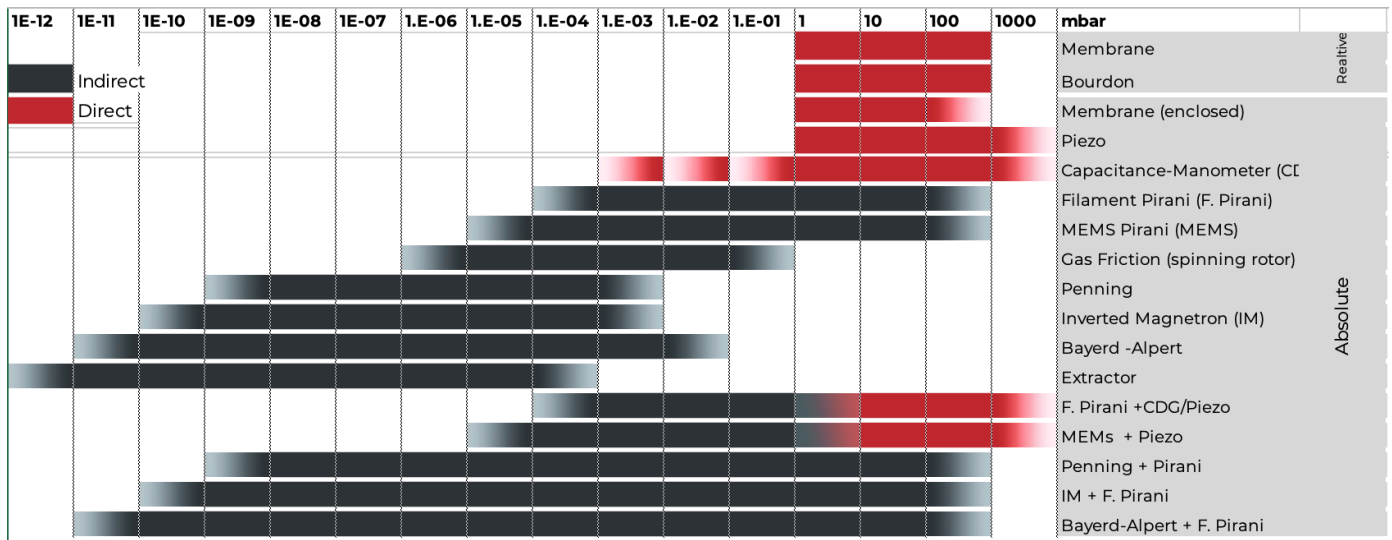| 1E-12	1E-11	1E-10	1E-09	1E-08	1E-07	1.E-06	1.E-05	1.E-04	1.E-03	1.E-02	1.E-01	1	10	100	1000	mbar	
																Membrane	Realtive
	Indirect															Bourdon	
	Direct															Membrane (enclosed)	
																Piezo	
																Capacitance-Manometer (CD	
																Filament Pirani (F. Pirani)	
																MEMS Pirani (MEMS)	
																Gas Friction (spinning rotor)	
																Penning	Absolute
																Inverted Magnetron (IM)	
																Bayerd -Alpert	
																Extractor	
																F. Pirani +CDG/Piezo	
																MEMs + Piezo	
																Penning + Pirani	
																IM + F. Pirani	
																Bayerd-Alpert + F. Pirani	

Figure 4.2: Operating range of different gauges.

Mechanical dial gauges are used in the pressure range from atmosphere to 1 mbar(a); however, these gauges offer only limited accuracy and can only be read directly at the point of installation. Diaphragm vacuum gauges (piezo sensors or capacitive sensors) are used to obtain more accurate measurements and for remote display of readings.

Pirani thermal conductivity vacuum gauges are used between 10 and 10^{-4} mbar(a). It is also possible to use special high-pressure hot cathode ionization vacuum gauges at pressures < 10^{-2} mbar(a).

Either cold cathode ionization vacuum gauges or hot cathode ionization vacuum gauges are used for pressures of less than 10^{-3} mbar(a), the latter in the case of clean conditions and rigorous accuracy requirements. It is also a good idea to use a combination of two or more sensors to cover the entire pressure range in use.

In the case of diaphragm vacuum gauges and Pirani vacuum gauges, pressure switch points are generated to activate ionization vacuum gauges only if the pressure is sufficiently low, thus protecting them against contamination or burn-out of the hot, thermionic, cathode. An overview on the operating range of different gauges is given in Fig. 4.2.

4.4 ACTIVE OR PASSIVE

Vacuum gauges can be either active or passive. "Active" gauges (as shown in Fig. 4.3) incorporate the electronic sensor, which transforms the pressure measured into an electrical signal with a compact electronic control and evaluation unit. They indicate the pressure measured either in the form of an analogue measurement signal or a numerical value through a standardized digital interface.

A "passive" gauge (as shown in Fig. 4.4) does not feature an integrated electronic unit, so they need to be connected to control units by a cable.

Figure 4.3: APG200 active pirani gauge.

This control unit will deliver power to the gauge head, evaluate the transmitted data and the evaluation and display the resulting pressure reading.

The control units are normally also equipped with analogue voltage outputs, digital interfaces and relay contacts for switch-points.

Figure 4.4: PRG20K passive pirani gauges.

4.5 DIRECT GAUGES

Elastic element vacuum gauges: A sealed and evacuated vacuum chamber is separated by a diaphragm from the vacuum pressure to be measured. This serves as the reference quantity. With increasing evacuation, the difference between the pressure which is to be measured and the pressure within the reference chamber becomes less, causing the diaphragm to flex. This movement may be transferred by mechanical means to a dial, or electrically by means of a strain gauge or a bending bar for conversion into an electrical measurement signal. Different types of elastic element gauges are shown in Fig. 4.5.
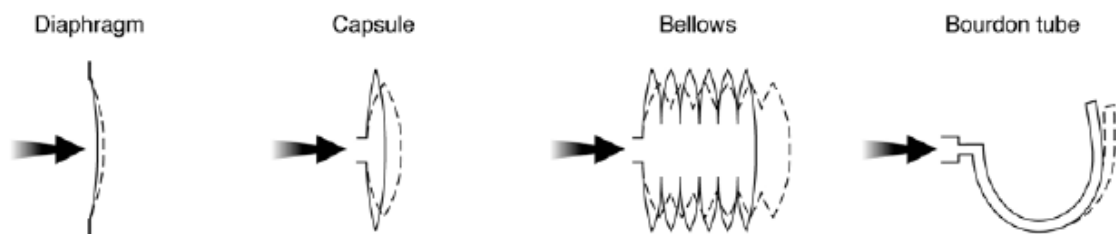

Figure 4.5: Types of elastic element gauges.

Bourdon vacuum gauge: The simplest direct gauges fall under the category of dial gauges. The most common is a Bourdon vacuum gauge that derives from the inside of a tube that is bent into a circular arc and connected to the vacuum system. Because of the external atmospheric pressure, the end of the tube bends more or less during the evacuation process. This actuates the pointer arrangement which is attached to this point. The corresponding pressure can be read off on a linear scale. With Bourdon gauges it is possible to roughly determine pressures between 10 mbar(a) and atmospheric pressure.

Capsule vacuum gauge:
This vacuum gauge contains a hermetically sealed, evacuated, thin-walled diaphragm capsule that is located within the instrument (see Fig. 4.6). As the vacuum pressure reduces, the capsule bulges. This movement is transferred to a dial via a system of levers and can then be read off as the pressure on a linear scale. It is independent of atmospheric pressure changes.
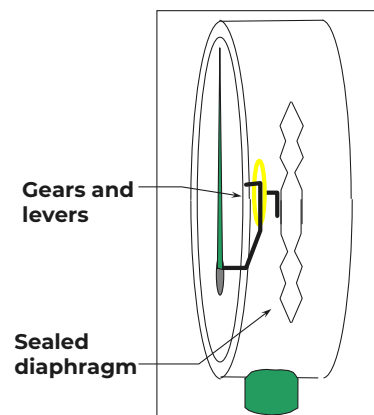

Figure 4.6: Schematic of a capsule vacuum gauge.

Capacitance diaphragm gauge (CDG): The term capacitive measurement means that a plate capacitor is created by the diaphragm with a fixed electrode behind the diaphragm. A schematic of a CDG is shown in Fig. 4.7. When the distance between the two plates of this capacitor changes, a change in capacitance will result. This change, which is proportional to the pressure, is then converted into a corresponding electrical measurement signal. An evacuated reference chamber serves as the reference for the pressure measurements. With capacitance gauges it is possible to accurately measure pressures from 10^{-5} mbar(a) to well above atmospheric pressure, whereby different capacitance gauges having diaphragms of different thickness, and therefore sensitivity, will have to be used.
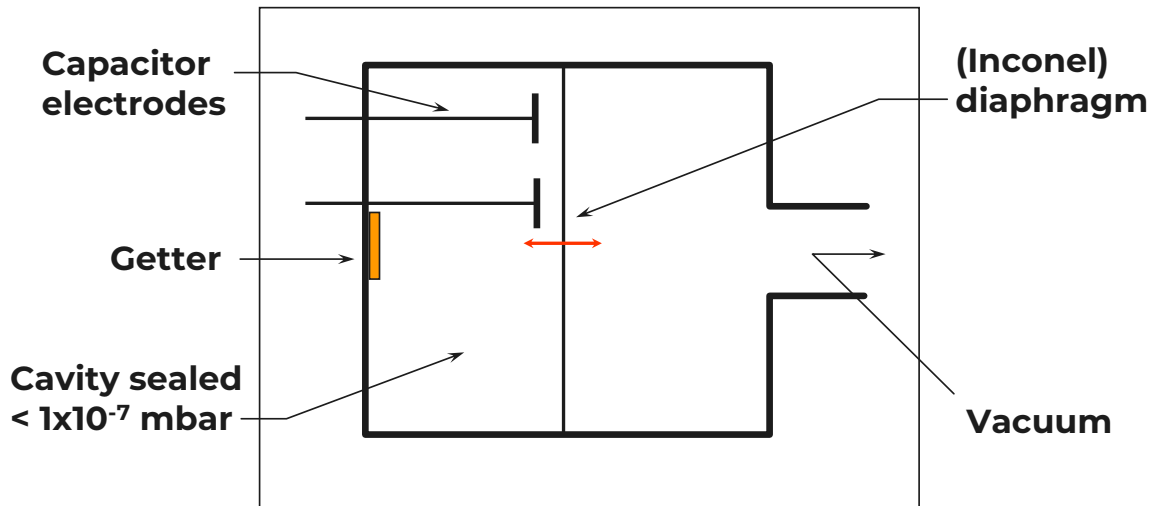

Figure 4.7: Schematic of a CDG.

Pirani gauge: The Pirani, or thermal conductivity gauge, uses the thermal conductivity of gases to measure pressure in the range from 10^{-4} mbar(a) to atmospheric pressure. The filament within the gauge head forms one arm of a Wheatstone bridge (see Fig 4.8).
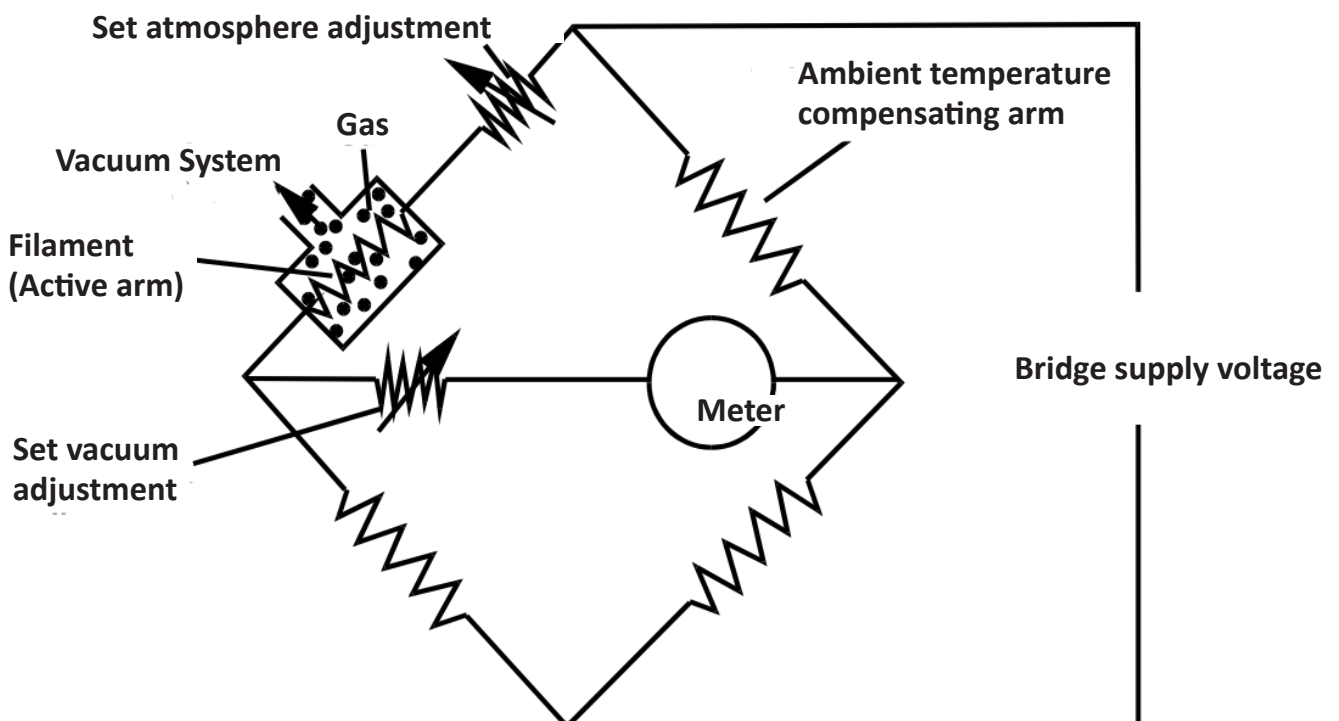

Figure 4.8: Schematic of a pirani gauge with a Wheatstone bridge.

In one operating mode the heating voltage which is applied to the bridge is controlled in such a way, that the filament resistance and thus the temperature of the filament remains constant regardless of the quantity of heat given off by the filament. Since the heat transfer from the filament to the gas increases with increasing pressures, the voltage across the bridge is a measure of the pressure (see Fig. 4.9).
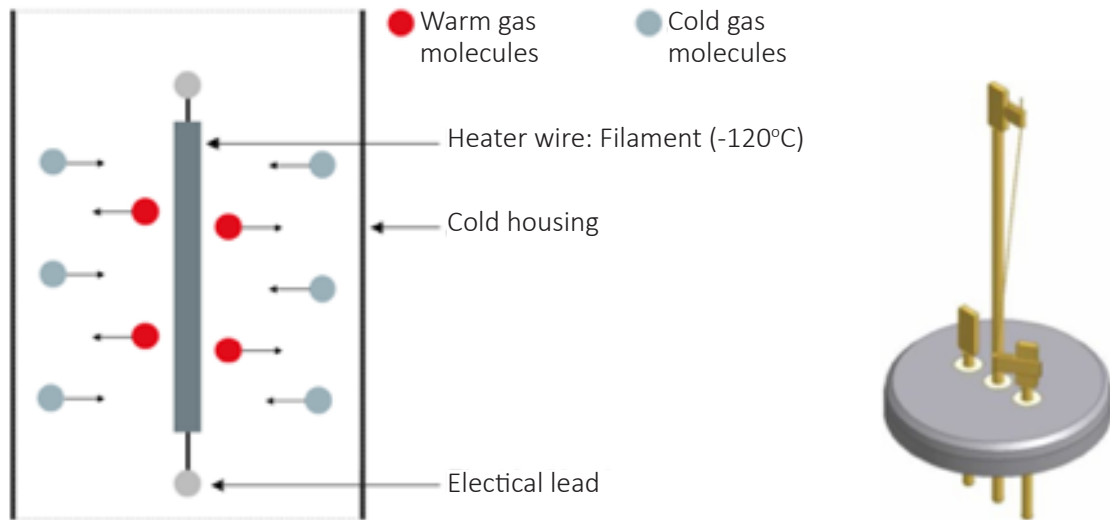
Figure 4.9: Operating principle (left) and set-up (right) of a pirani gauge.

The pirani gauge is an indirect gauge since thermal conductivity varies with gas species as shown in the calibration chart in Fig. 4.10.
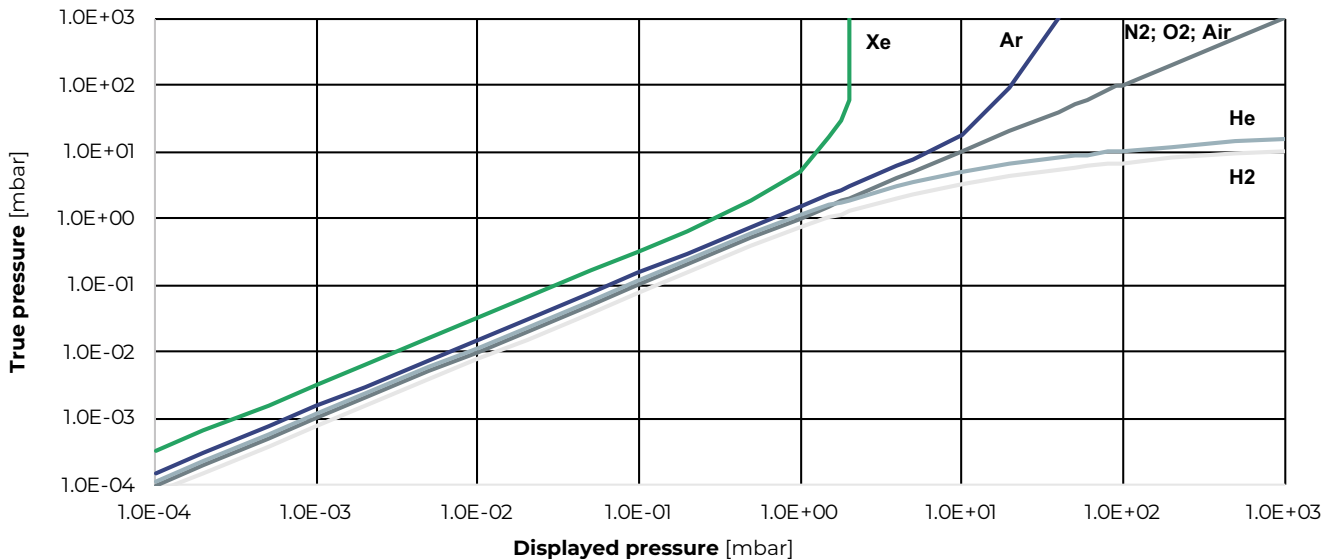
Figure 4.10: Gas dependency of thermoconductivity gauges.

Hot cathode ionisation vacuum gauge: this gauge emits electrons from a heated cathode which are attracted to a positively charged anode. During their flight they ionize gas molecules; the ion current produced being proportional to the gas pressure.

The hot cathode sensors which are mostly used today, are based on the Bayard-Alpert (BA) principle. A BA vacuum gauge collects positive ions on a thin ion collector wire (see Fig. 4.11) and measures the resulting current to the ion collector to determine the number of

molecules present, and indicates a pressure based on that measurement. The Bayard-Alpert gauge was invented by R.T. Bayard and D. Alpert in 1950 to overcome a limitation in vacuum pressure measurement by the triode gauge of ~10^{-8} mbar(a).

Grid Anode (+ve)   **Filament Cathode**

Ion Collector

Figure 4.11: Schematic of a BA gauge.

This is because electrons striking the grid anode create low-energy X-rays, which emit photoelectrons when they strike the ion collector. The current that results from the photoelectrons leaving the collector is what causes the l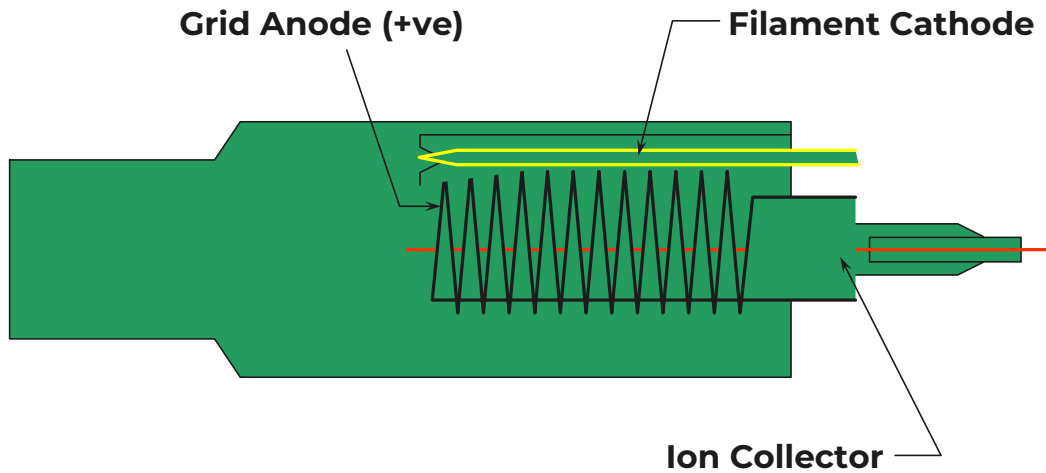ower pressure limit of the triode gauge. The solution proposed by Bayard and Alpert was to reconfigure the collector and grid of the triode gauge to lower the current from this X-ray effect.

● Gas   ● Ion +   ● Electron-

Emission current →   Ion current →

Cathode
50 Volt

Anode
200 Volt

Ion collector
0 Volt
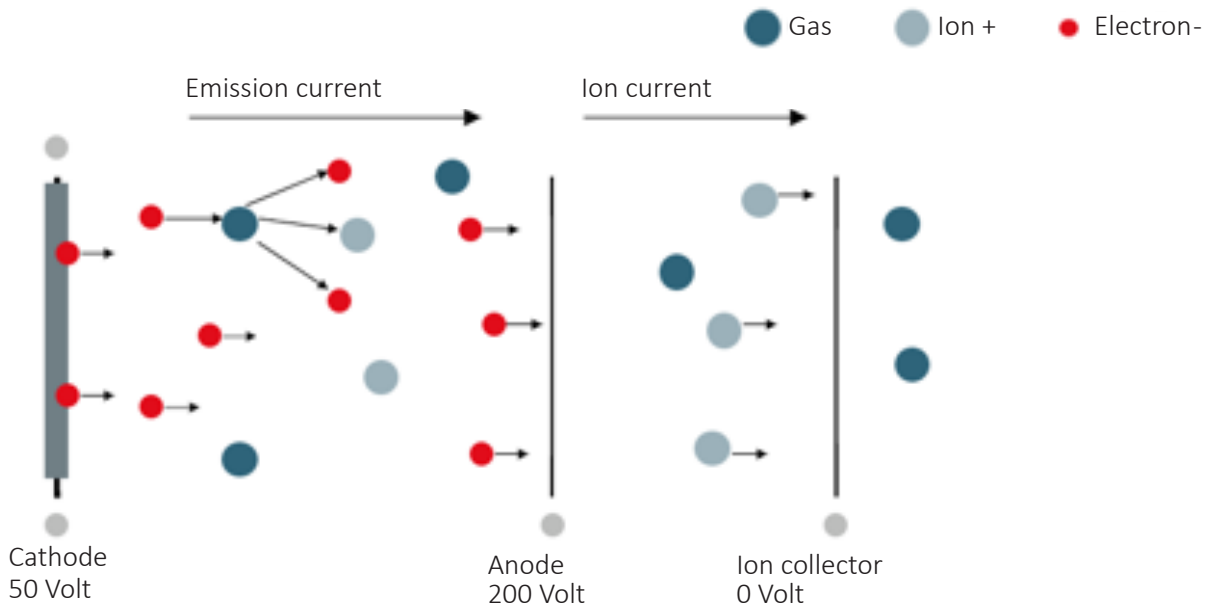
Figure 4.12: Operating principle of extractor ionization gauge.

With the modern electrode arrangement, it is possible to make measurements in the pressure range from 10^{-10} to 10^{-4} mbar(a); other electrode arrangements permit access to a higher pressure of 10^{-2} mbar(a). For the measurement of pressures below 10^{-10} mbar(a), so-called "extractor" ionization gauges are employed in which ions are focused onto a very thin and short ion collector (see Fig 4.12). Due to the geometrical arrangement of this system, interfering influences such as X-ray effects and ion desorption can be almost eliminated. The extractor ionization gauge permits pressure measurements in the range from 10^{-4} to 10^{-12} mbar(a).

Cold cathode ionization gauges: A cold cathode ionization gauge is so named as the cathode is not heated and commonly operates on the inverted magnetron principle. In this device a gas discharge is ignited by applying a high voltage. The resulting ion current is output as a signal which is proportional to the prevailing pressure. The gas discharge is maintained at low pressures with the aid of an applied magnetic field.

A cold cathode system requires a high ignition voltage of up to 3.5 kV DC. With this high acceleration voltage, the electrons fly on the shortest path to the positive anode ring or pin. The electrons are forced into tight spiral paths by the directed permanent magnetic field. This considerably extends the residence time of the electrons, which also considerably increases the probability of the ionization of gas atoms. This creates a permanent ion current whose size is proportional to the pressure.

While the cold cathode ionization gauge uses a closed magnetic field in the sensor, another variant of cold cathode gauge uses an outwardly open magnetic field as an inverted magnetron. This is always indicated from the outside by a warning sign for pacemakers.

Although applications are widely varied, the basic parameters for choosing a gauge are similar. Most users want the most accurate reading possible and settle for a gauge that covers the ranges that are important to them. There are a lot of different vacuum gauge types available, but with a full understanding of the required application, it should be possible to select the right gauge and controller for any operation.

5. VACUUM LEAK DETECTION

5.1 CREATING A LEAK TIGHT SYSTEM

Wherever and whenever a vacuum needs to be created, it is essential to ensure as far as possible, the integrity (*i.e.* the leak-tightness or simply "tightness") of the system. If the system is not tight, then both time and effort are pointlessly squandered trying to create and maintain a vacuum in an "open system" which could never support a vacuum in the first place. Of course, this is an extreme case, but as vacuum pressures get lower and lower, even the most seemingly secure and pristine of systems will soon show that they are less than tight.

Leaks can appear in various ways and their effect differs based on their size and form. A leak allows the gas to transport from one place to another. As gas is compressible, the pressure of the gas influences the amount of the transported gas. That is why the leak rate is always specified in mbar(a)l/s or equivalent units.

Being able to produce a system which is completely devoid of any leaks is virtually impossible and, therefore, it is necessary to work towards achieving a leak rate that is acceptable to the application in question, *i.e.* "sufficiently" leak-tight.

Tightness (or "the absence of leaks") is required for numerous reasons, including:

- for operator safety (*e.g.* egress of toxic materials)
- to guarantee a long lifetime of products
- to ensure and maintain the pressure/vacuum
- to guarantee product safety (*e.g.* to stop air entering a system to create an unsafe mixture)
- for environmental and/or quality standards
- and for process efficiency.

There are multiple ways of detecting vacuum leaks, which are shown in Table 5.1.
The method to be employed for detecting and measuring leaks depends on the lowest likely detectable leak rate required. The leak rate is the amount of gas that flows through a leak at a given pressure differential per unit time.

mbar	10^{-12}	10^{-11}	10^{-10}	10^{-9}	10^{-8}	10^{-7}	10^{-6}	10^{-5}	10^{-4}	10^{-3}	10^{-2}	10^{-1}	10^{0}	10^{1}
Bubble Test									←———————————————————————→					
Differential pressure										←—————————————————————→				
Pressure Decay Test										←—————————————————————→				
Pressure Rise Test							←——————————————————————————————————→							
Helium Sniffer Mode						←——→								
Helium Vacuum Mode		←——→												

Table 5.1: Different methods of leak detection and their application range.

Leak testing generally aims to identify the position of a leak and/or measure the flowrate of gas through a leak.

5.2 TYPES OF VACUUM LEAK DETECTION

Bubble and foam-spray tests

There is no single detection method suitable for every eventuality. Which one to employ is usually based upon the pressure/vacuum range and the system under examination. The most simple is the bubble test, which is best illustrated by placing a punctured bicycle tube under water, trying to inflate it, and noting where the bubbles come from.

A slight variation of this, the foam-spray test, involves brushing or spraying a surfactant or liquid soap solution around the joint/connection of an active water/gas pipe, and observing if the liquid forms a foam. Both the bubble and foam-spray test are reliable ways of detecting a leak, however, they are time consuming and depend on the attentiveness of the inspector.

Additionally, whilst simple and effective at detecting the locations of leaks, quantifying a leak rate measurement is not practical without a great deal of additional equipment.

Pressure rise tests

The pressure rise tests are conducted by:

A. Evacuating a closed vacuum vessel under test until a certain pressure is obtained, then closing the pump's inlet valve. Fig. 5.1 shows the schematic set-up. After a pre-determined period of time, the inlet valve is again opened, and the time is recorded for the pump to return the pressure to the original evacuated level. This process is repeated a number of times. If the time to return the pressure to the original level remains constant, then a leak is present. If this time period decreases, this indicates reduced gas liberation (outgassing) on the inside of the system (a "virtual" leak) on each successive pump-down but does not exclude a leak from being present.
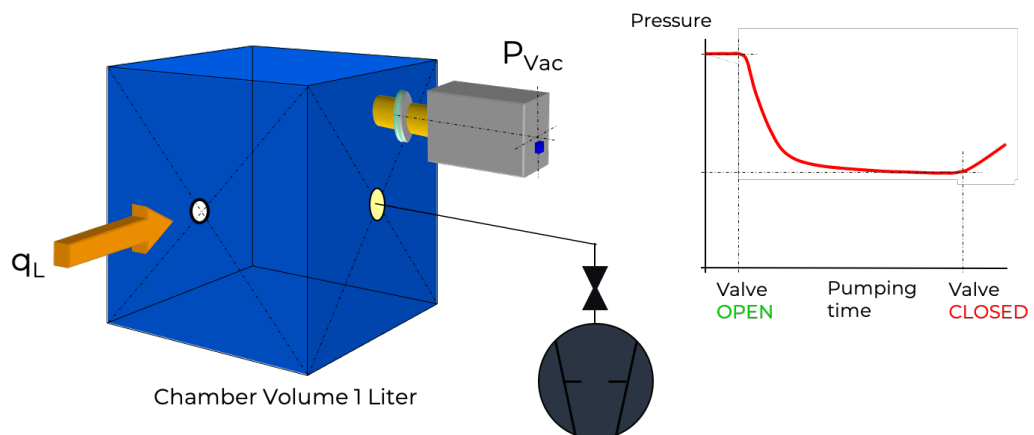

1. Pump down starts with valve open, P_{Vac} decreases
2. Valve closes, P_{Vac} should be stable
3. The leak q_L delivers a continuous gas flow into the chamber
4. P_{Vac} increases with valve closed

Figure 5.1: Schematic set-up for a pressure rise test.

B. Then plot the pressure against time for an evacuated and then isolated system. The curve for the rise in pressure will be a straight line if a leak is present and will tend to atmospheric pressure. However, if the pressure rise is due to gas liberation from the system walls, the pressure rise will gradually taper off to reach a final, stable value (refers to Fig. 5.2).

In most instances both phenomena occur simultaneously, which makes separating one from the other almost impossible. If the volume of the chamber or item under test is known, then the leak rate can be calculated as the volume x (measured change in pressure)/time taken.
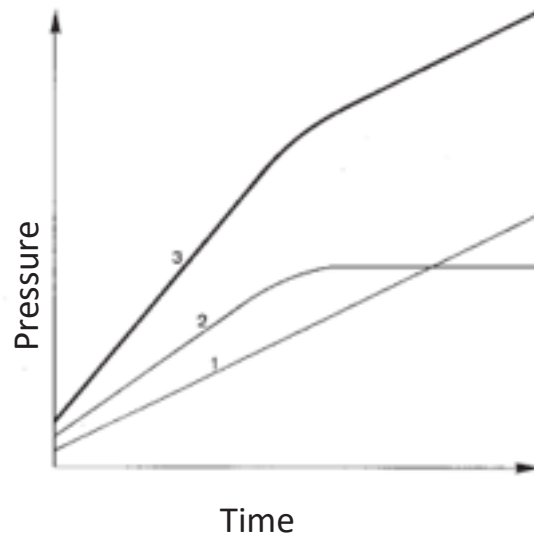
1. Leak
2. Gas evolved from the container walls
3. Leak + gas evolution

Figure 5.2: Pressure development for an evacuated and isolated system.

Pressure drop test

This test is not dissimilar to the pressure rise test. It is only rarely used to check leaks in vacuum systems, and only when the (positive) gauge pressure does not exceed 1 bar, since the flange connections used in vacuum technology will not tolerate a higher pressure.

However, the pressure drop test is frequently employed in tank engineering. Pressure drop tests allow leak rate measurements to 10^{-4} mbar(a)l/s but can be distorted if condensation occurs.
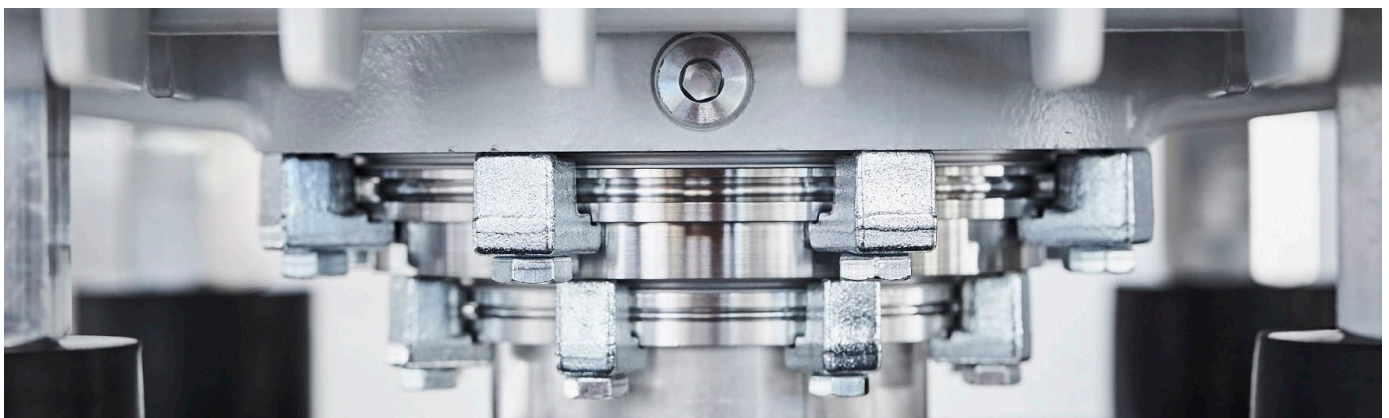
Helium leak testing

Other leak test methods are employed but the only method to detect leaks smaller than 1×10^{-6} mbar(a)l/s is with a helium leak detector (HLD). The most accurate and rapid leak detection methods employ helium as the tracer gas, coupled with a mass spectrometer for identification and quantification typically to minimum detectable leak of 10^{-12} mbar(a)l/s.

The reasons for using helium (mass, $M = 4$) for leak detection are compelling: It is unequivocally detectable by a mass spectrometer, it is chemically inert, non-explosive, non-toxic, present in normal air in a concentration of only 5 ppm, and is relatively economical.
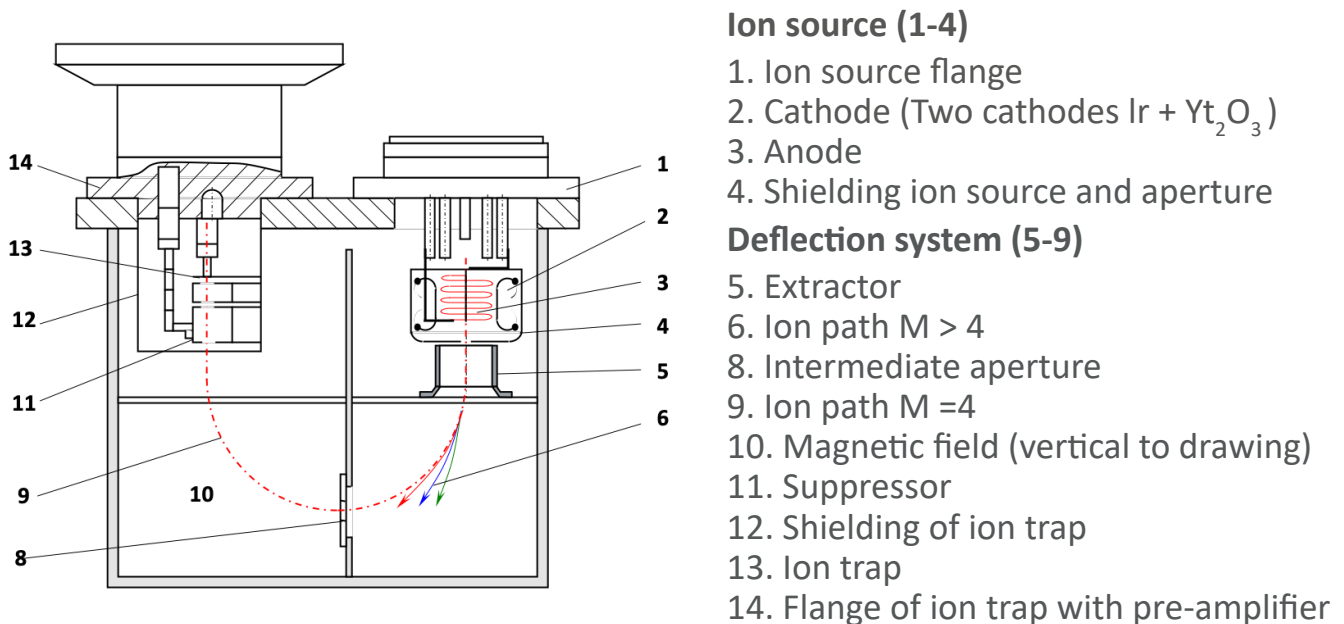

Ion source (1-4)

1. Ion source flange
2. Cathode (Two cathodes Ir + Yt_2O_3)
3. Anode
4. Shielding ion source and aperture

Deflection system (5-9)

5. Extractor
6. Ion path M > 4
8. Intermediate aperture
9. Ion path M =4
10. Magnetic field (vertical to drawing)
11. Suppressor
12. Shielding of ion trap
13. Ion trap
14. Flange of ion trap with pre-amplifier

Figure 5.3: Schematic of a He leak detector.

Leak detection using helium: working principles

Leak detection using helium is achieved in the following ways: The unit being checked is either pressurised from within with helium or else pressurised/sprayed from without with helium. The gases from any potential leaks are collected and pumped into the mass spectrometer for analysis, and any value above the background trace of helium is evidence of a leak. The mass spectrometer is by far the most sensitive leak detection method, as well as the one most widely used for detecting the smallest leaks. In principle, it is possible to detect all gases using mass spectrometry, however, helium as a test gas has proved to be especially practical.

The mass spectrometer itself works in the following way: any helium molecules entering into the spectrometer will be ionized, and these helium ions will then travel into the ion trap, where the resulting ion current is analysed and measured. Based on the ionization current, the leak rate can be calculated. Fig. 5.3 shows a schematic of a He leak detector.

The reference (or background) reading for helium is a crucial part of the leak measurement procedure. This reference reading is the "background noise" for helium, which can be
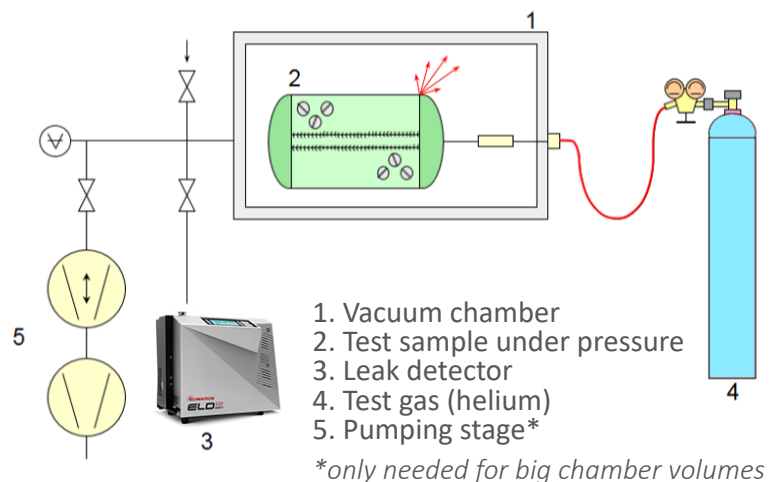
thought of as the ambient level of helium in the air.

The background reading can be influenced and constituted by the increases in the ambient helium concentration (from the act of using helium itself), helium being trapped and permeated from polymers or a memory/clean-up effect in the HLD itself. Modern HLDs constantly measure and calculate this internal (background) level and automatically subtract this from the leak rate measurement.

Helium leak detection measurement methods

There are two HLD methods: integral testing and local testing. The choice of which method to use depends on the configuration and leak rate to be measured too. The "integral" method shows if there is a leak present whilst the "local" method shows where a leak is located (however, the exact pin-pointing of the leak location, is difficult). Both of these detection methods can each be sub-divided down into two further parts: "sample under pressure", and "sample under vacuum".

The first of the two integral testing procedures is called the **"integral (sample under pressure)" method**, with the chamber under investigation being placed in a sealed container. The chamber (or sample under test) is pressurised up with helium and the container is connected to the leak detector. A sample of gas from within the container is drawn and passes through the mass spectrometer where any increase (over the background reading) in helium levels is indicative of a leak.

1. Vacuum chamber
2. Test sample under pressure
3. Leak detector
4. Test gas (helium)
5. Pumping stage*

only needed for big chamber volumes

In the **"integral testing (sample under vacuum)" method**, the chamber is again placed within a container but in this instance the container is pressurised with helium – and the test chamber is directly connected to the leak detector. A sample of the gas within the chamber is drawn off and passes through a mass spectrometer where again, any increase in helium (above the background reading) is indicative of a leak. Fig. 5.4 shows the general set-up for the two methods.

1. Pressure chamber
2. Test sample under pressure
3. Leak detector
4. Test gas (helium)
5. Pumping stage*
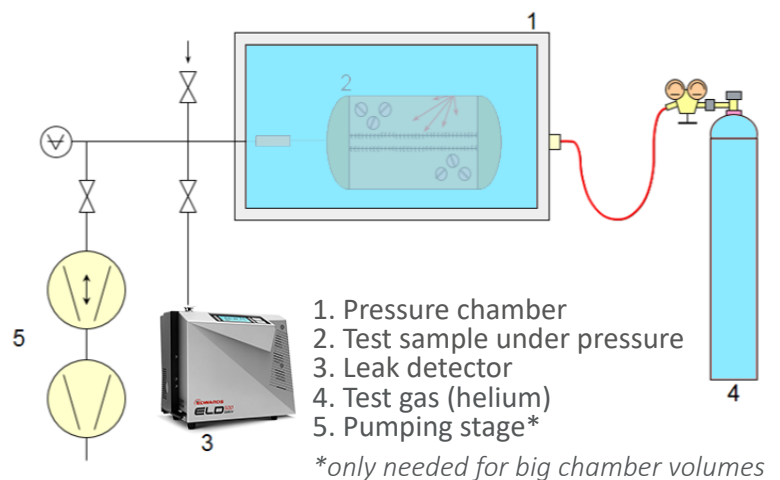
only needed for big chamber volumes

Figure 5.4: Helium leak detection methods. "Sample under pressure" (top) and "sample under vacuum" (bottom).

Sniffer or spraying testing

The second set of procedures are sometimes referred to as the "sniffer" or "spraying" tests. In the **"local-sniffer (sample under pressure)" method**, the chamber is pressurised with helium and a sniffer device is passed around the chamber's likely leak points (welds, flanges, portals, instrument ducts etc.) to capture any escaping tracer gas. This "sniffed" gas is passed to a mass spectrometer to record any elevated (*i.e.* above background) helium levels (see Fig. 5.5).
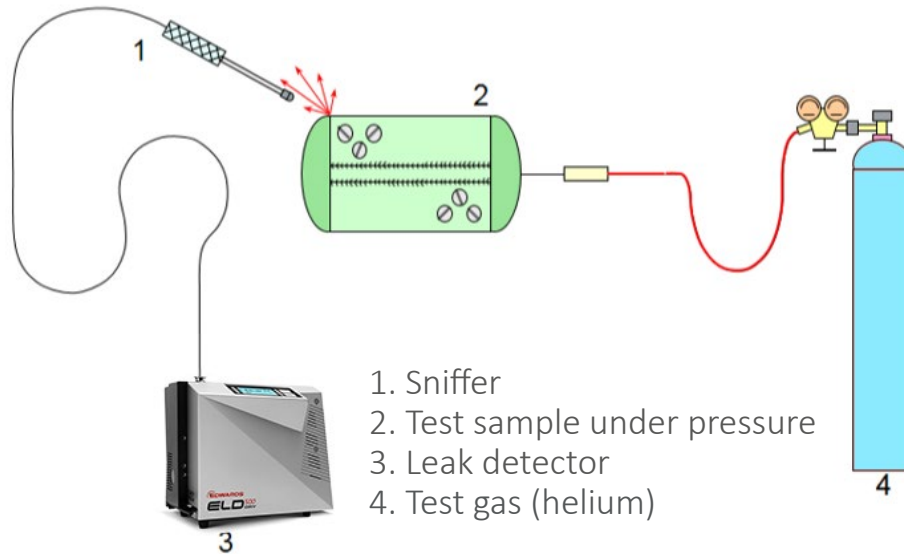

1. Sniffer
2. Test sample under pressure
3. Leak detector
4. Test gas (helium)

Figure 5.5: Schematic set-uo the "local-sniffer" method.

In the **"local-spraying (sample under vacuum)" method**, the chamber is vacuum pumped and helium gas is liberally spray-directed towards likely leak points, with the intention that some of this pure helium will be transported into the chamber (see Fig. 5.6). The gas, from within the chamber, is passed into a spectrometer to record any elevated helium levels.

1. Test gas sprayer
2. Test sample under vacuum
3. Leak detector
4. Test gas (helium)
5. Pumping stage*

only needed for big chamber volumes
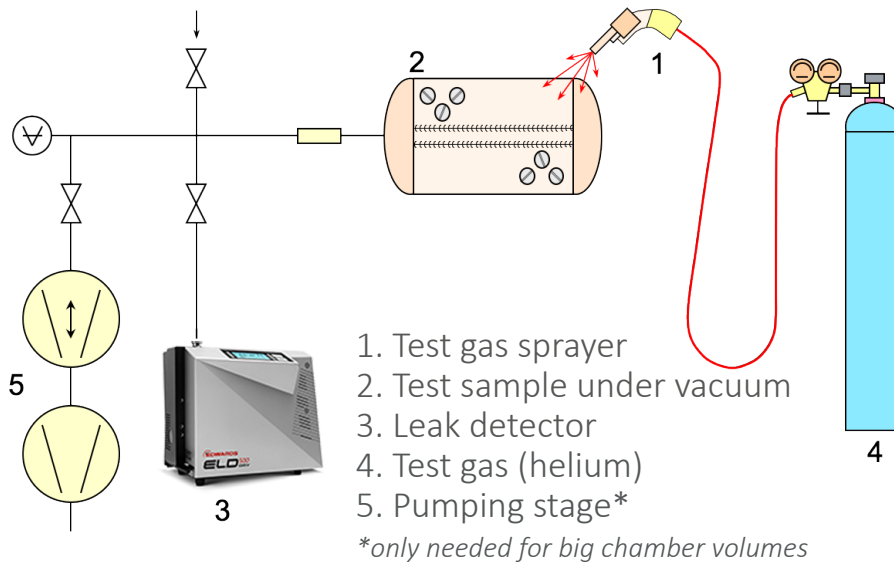
Figure 5.6: Schematic set-up the "local-spraying" method.

In summary the HLD integral method requires the chamber or part under test to be placed inside a leak-tight unit (which is not always a possibility). In contrast in the local testing method the chamber is either internally pressurised with helium or internally evacuated and helium being sprayed onto the surface of the chamber at suspected leak points. In both tests, helium enters the leak detector via possible leaks and passes to the spectrometer for analysing.

6. APPLICATION OF VACUUM TECHNOLOGY

6.1 WHY USE VACUUM?

Product quality and yield improvements, reductions in operating costs (especially energy costs) and the compliance with ever restrictive national and international environmental regulations, particularly with respect to waste water and effluent disposal, require chemical processes to be re-appraised. More and more processes are, therefore, operated under reduced and ever reducing pressure conditions. Over the past years steam ejectors, the hitherto favourite vacuum production means in the process industry, have been replaced in succession by steam ejector + liquid ring pump combinations, by mechanical booster liquid ring pump combinations and once-through oil-lubricated rotary pumps. The latest trends towards minimum efflluent production now favours all mechanical pumping such as roots-rotary and roots-dry pump combinations with some form of inlet, interstage and exhaust condenser.

Fundamentally all chemical processes are based on some form of simultaneous heat and mass transfer, the interaction of which differentiates the various chemical processes from each other. For example, if a substance is subjected to a lower than ambient pressure or equilibrium pressure, mass transfer in the form of evolving solvent vapour will occur to create a new natural equilibrium level. To evolve vapour or to evaporate a solvent, energy in the form of heat must be available. If no heat is added the heat of evaporation will be taken from the immediate surroundings creating a cooling effect, *i.e.* an evaporative cooling process. If on the other hand, heat is added in any form the resultant processes become one of drying for solids, distillation for liquids, crystallisation for combinations of solids and liquids etc.

Most vacuum processes within the pharmaceutical industry are operated in the pressure range of 20 to 10^{-3} mbar(a) and in that pressure range certain process adjustments will be promoted or enforced by the creation of concentration gradients which can be loosely termed as separation effects.

Most familiar is the lowering of the boiling point under vacuum, which permits the distillation of substances and the freeze-drying of biological materials etc. subject to chemical changes at the normal boiling point. These changes include oxidation, pyrolysis, molecular rearrangement, molecular condensation and polymerisation.

Vacuum is also important when processing heat-stable compounds. These have low vapour pressure characteristics and can therefore require excessively high temperatures to achieve the desired evaporation rates at atmospheric pressure. These substances may then be economically processed using an available low temperature heat source such as low pressure waste steam, or other recovered and/or heat pump upgraded heat transfer media.

Expressed in very general terms a reduction in pressure by one decade *i.e.* from say 10 to 1 mbar(a), will bring about a 40 to 50°C lower processing temperature and therefore a considerable reduction in energy requirements and costs.

6.2 ENVIRONMENTAL CONSIDERATIONS

Of late, environmental considerations are strongly influencing the choice of process parameters, process plant design and operation and in particular the correct and cost effective choice of the vacuum producing equipment. This trend will certainly continue to intensify.

Process water supply and waste water disposal costs are currently rising faster than any other. Water disposal charges in most developed countries, and Europe in particular, are based on the amount of contamination, the nature of contaminants and their natural oxygen requirements for safe and complete degradation. Low contaminated process water is generally discharged into waste mains, rivers, lakes or seas with responsibility for treatment or cleaning-up in the hands of the local Water Authority. This is generally the most expensive disposal method in developed countries.

In-house treatment of low contaminated waste as produced by steam ejectors and liquid ring pumps, can also attract additional costs which typically range from considerable to excessive. The current trend is towards the production of the most concentrated waste suitable for biological degradation and digestion, at considerably lower operating costs, with the intention of producing a saleable or reusable by-product. For this reason alone, mechanical vacuum producing equipment with indirect condensation and exhaust scrubbing devices is currently favoured.

Based on cost reductions, for primary heat energy and waste disposal, pay-back periods of less than eight months have been reported for mechanical vacuum pumping systems.

6.3 BASIC VACUUM PROCESS PLANT

Basic plant configuration comprises a vacuum process vessel with associated heat transfer and vapour condensation/separations means. Distillation applications, for example, have predominantly vertical cylindrical process vessels with heating jackets practically over the entire cylindrical section and internally arranged condensation surfaces. For drying applications a cubicle horizontal vessel with internally arranged heated product support shelves and external vapour condenser is most common.

Vacuum pumping systems are selected according to the required process pressure which is generally a function of the process temperature and the product's physical characteristics, especially its vapour pressure.

A typical basic system is shown in Fig. 6.1. Depending on the application and pump technology used, a pre-condenser might be used to capture condensable vapours before entering the vacuum pump. The vacuum pump system may comprise of a single pump unit or multiple pumps, depending on the pressure regime required. For a two- (or multi-) stage system, first stage compression is typically a mechanical booster stage. There may be a nitrogen purge/gas ballast flow added to help prevent condensation. If a two-stage system is used, an interstage condenser or simple knock-out pot (KOP) may be required to capture condensable vapours between the pump stages. The final stage vacuum pump may be an oil-sealed, liquid ring or mechanical dry pump. The discharge line from the pump exhaust typically connects to the shared exhaust line or an abatement system, like a scrubber.
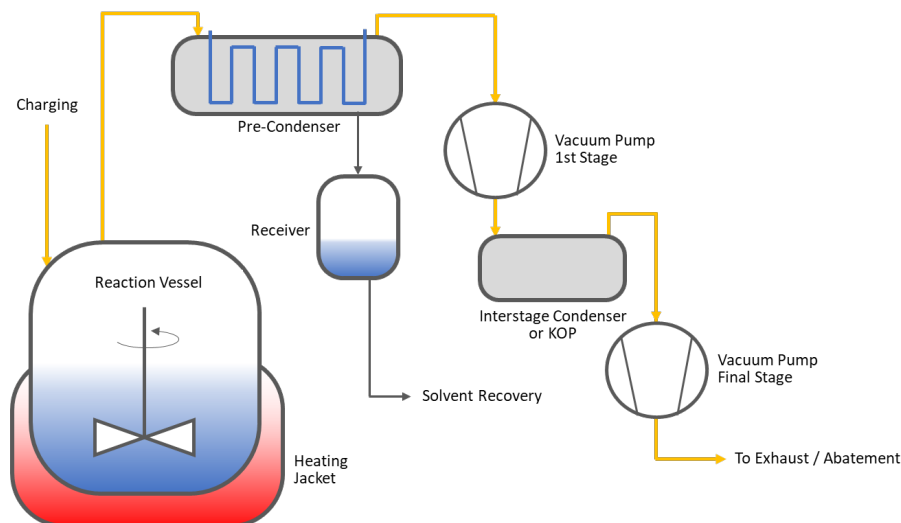
Figure 6.1: Basic vacuum system schematic.

Unfortunately, chemical processing involves materials which provide severe operating conditions for vacuum pumps and components. The materials to be handled are mainly vapours and gases which may be highly toxic, chemically aggressive and potentially explosive and solid carry-over may be present which may be highly abrasive or form solid deposits.

Each type of process produces its own set of problems and therefore requires careful independent consideration and solution(s). Although certain protective measures such as separators, filters, flame arrestors, condensers etc. both up and downstream of the pumps may be fitted, these do not totally overcome the contamination problem(s).

6.4 CHOOSING THE RIGHT PUMP FOR THE APPLICATION

For the chemical industry, Edwards offers a wide range of vacuum pumps and systems to meet the demanding process applications in base and fine chemicals, specialty chemicals and pharmaceutical applications. With rapid advances in technology, chemical processing industries place increasingly challenging demands on vacuum systems.
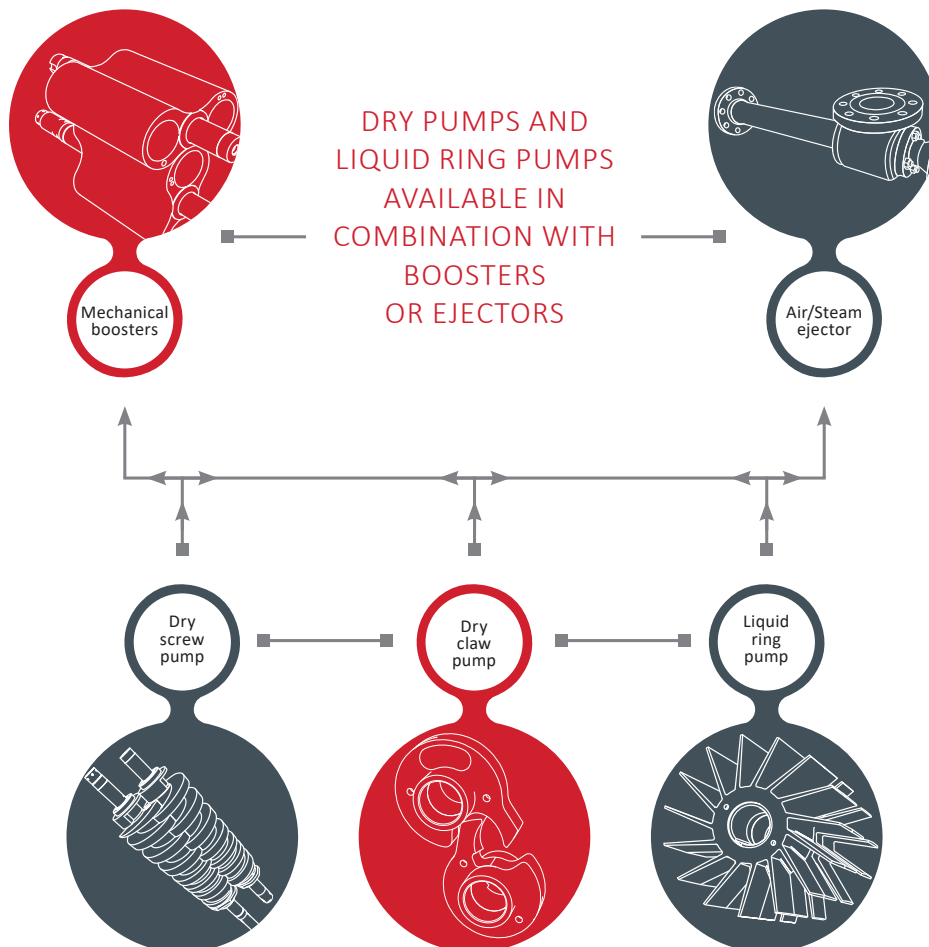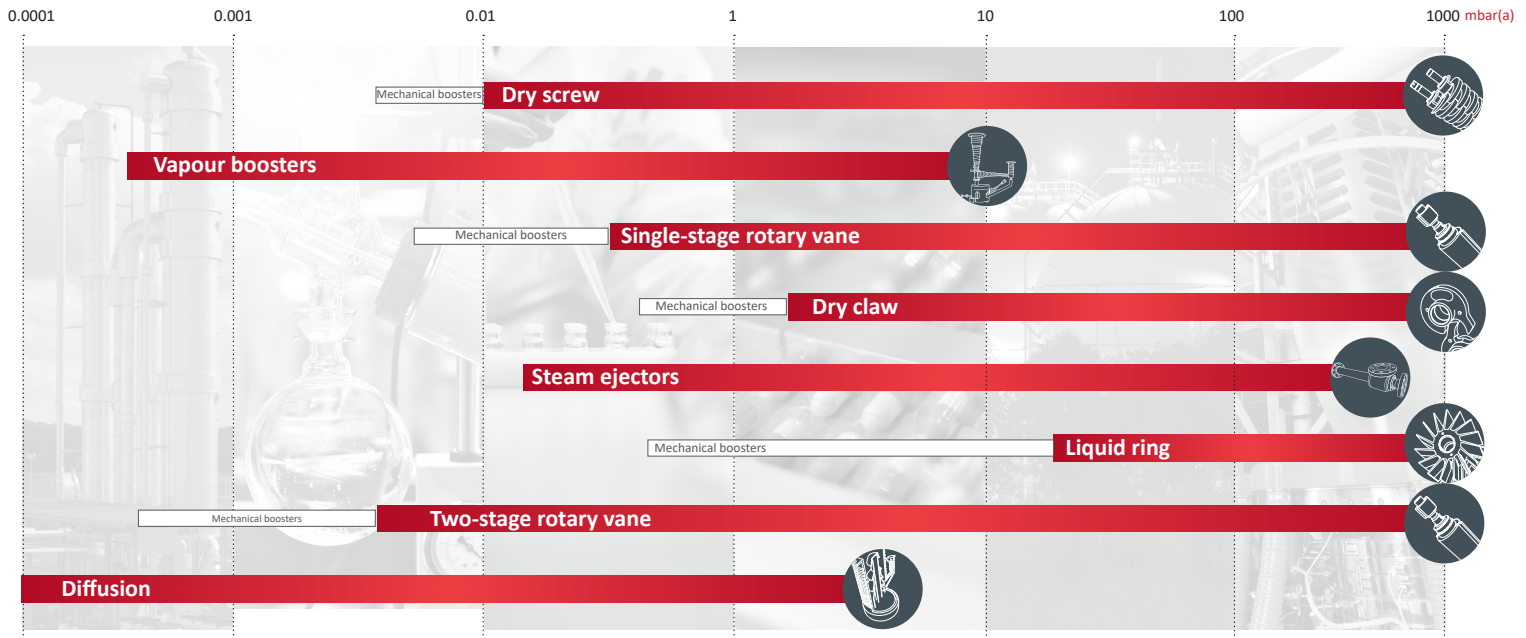
VACUUM ENABLES QUALITY PRODUCTION FOR A WIDE RANGE OF PROCESSES

Other Applications	Molecular Distillation	Fine Chemicals	Pharmaceuticals	Petrochemicals	General Vacuum

APIs | Adhesives | Aromatics | Basic chemical processing | Biofuels | Detergents

Fertilisers and pesticides | Fibre production | Flavours and fragrances | Olefins | Oleochemicals

Pharma end products | Pigments and paints | Refinery VDU | Resin and polymer production

Vacuum is used in a wide range of chemical processing and manufacturing applications. A vacuum system can affect every aspect of a process, such as utility consumption, processing times, quality, safety, and environmental impact.

The benefits of vacuum include:
- Reduced energy consumption
- Reduced risk of heat damage to sensitive products (lowering processing temperature)
- Enhanced product quality through impurity removal
- Safer operating atmospheres through oxygen exclusion
- Increased yield by shifting reaction equilibrium

The design of the vacuum system, including pumps, piping, controls, and other accessories, is determined by process parameters and evacuation requirements. Correct sizing is vital to ensure that the processing objectives, such as vacuum pressure, pumping speed, and budgets, are met. The most important factors in the choice of vacuum producing equipment include cleaner vacuum, reliability and lower cost of ownership.

Vacuum system design considerations:
- Pressure, temperature, process vapour types and flowrate
- Volume to evacuate and operating point(s)
- Utilities available and costs
- Flammability & other hazards – explosion and safety issues - ATEX
- Design pressure and temperature
- Vapour, liquid and solids handling
- Reliability, flexibility, maintenance
- Process cleanliness
- Environmental impact
- Cost of ownership
- Support & service

APPLICATION CHART

	Chemical Dry Pump	Mechanical Booster	Rotary Piston Pump	Rotary Vane Pump	Liquid Ring Pump	Steam Ejector
Distillation, normal	✓ ✓	✓ ✓	✓	✓	✓ ✓	✓ ✓
Short path distillation	✓ ✓	✓ ✓	✓	✓		✓ ✓
Molecular distillation	✓ ✓	✓ ✓	✓	✓	✓	✓ ✓
Reactor service	✓ ✓	✓ ✓	✓		✓ ✓	✓
Central vacuum (flammables and corrosives)	✓ ✓	✓ ✓			✓ ✓	
Biofuels	✓ ✓	✓ ✓			✓ ✓	✓ ✓
Drying, evaporation, crystallisation, concentration	✓ ✓	✓ ✓	✓	✓	✓	✓
Gas recovery / recirculation	✓ ✓	✓ ✓				
Degassing	✓ ✓	✓ ✓	✓	✓ ✓	✓ ✓	✓
Absorption, adsorption, desorption	✓ ✓	✓ ✓			✓ ✓	
Pervaporation	✓ ✓	✓ ✓			✓ ✓	
Solvent recovery	✓ ✓	✓ ✓			✓	
Isocyanates production	✓ ✓	✓ ✓		✓	✓ ✓	✓ ✓
Impregnation	✓ ✓	✓ ✓	✓ ✓	✓ ✓	✓ ✓	
Polymers and plastics production	✓ ✓	✓ ✓	✓	✓	✓ ✓	✓ ✓
Paint, pigments, coatings and ink production	✓ ✓	✓ ✓	✓	✓ ✓	✓	✓
Soaps / detergents production	✓ ✓	✓ ✓	✓			✓ ✓
Ethylene Oxide sterilisation	✓ ✓	✓ ✓		✓ ✓		
Oil treatment plants	✓ ✓	✓ ✓	✓ ✓	✓	✓	✓
Dewatering and filtration	✓ ✓	✓ ✓			✓ ✓	
Flammable and corrosive gases	✓ ✓	✓ ✓		✓	✓ ✓	✓ ✓

✓ ✓ Preferred technology or widespread use   ✓   Some applications

	Lowest cost of ownership		High cost of ownership typically		Highest cost of ownership typically

6.5 PUMP SIZING

The size of the vacuum pump or vacuum pump system is governed by two independent questions:

1. What effective pumping speed is needed to pump down a given volume of a vessel or reactor to a desired pressure in a given time?

2. What effective pumping speed is needed during a vacuum process with a given load of gases or vapours to maintain the desired operating pressure?

The effective or net pumping speed of a vacuum pump is thereby defined as the actual pumping speed that prevails at the vessel taking into account the flow resistance (conductance) of any baffles, condensers or catch pots, valves, filters and pipes installed between the pump and the vessel (for calculating the net pumping speed refer to Sec. 2.2, equation (2-9)). Depending on the system design, the effective pumping speed can differ significantly from the nominal pumping speed of the vacuum pump.

6.5.1 AIR LEAKAGE

Leak tightness is a significant factor when sizing a vacuum system. The gas load of non-condensables is often underestimated. Air entering the system through leakage is usually the most significant load of non-condensables in a vacuum process. This may result in a significantly over-sized vacuum system, due to:

1. The required capacity of the system being increased by the amount of air that needs to be pumped "away" to maintain the process pressure. This will be reflected in capital costs as well as running costs.

2. The efficiency of condensers being reduced as the air passing through the condenser will be saturated with process vapours, thus increasing the total gas load.

3. Vapour emissions from the system being increased. Depending on the solvents used, this may be subject to environmental concerns.

Therefore, minimizing the load of non-condensables and air resulting from leakage can have a significant impact on process efficiency and costs.

To minimize air leakage the following points should be considered:

1. High quality process vessels should be used for vacuum duties.

2. Equipment connections should be minimized and all-welded connections should be used where possible.

3. Equipment seals should be inspected regularly and replaced whenever deterioration is observed.

4. The air leakage should regularly be determined by measuring the rate of pressure rise (refer to chapter 5.2 on how to do a pressure rise test).

Once the rate of pressure rise has been determined, the air leakage can be estimated according to BS3636 by using the formula:

$$L = 0.07 \cdot V \cdot \Delta p \qquad (6\text{-}1)$$

where:

L = leak rate [kg/h],

V = total volume of the system to be evacuated, including pipes, receivers, etc., [m³],

Δp = pressure rise, [mbar(a)/min]

Typically, a pressure rise of 2-3 mbar(a)/min is achieved on existing plants and 0.75-1.5 mbar(a)/min for new plants.

Air from leakage may not be the only source of non-condensables. For specific purposes air or nitrogen is deliberately introduced to the system to prevent condensation, control the pressure, improve the vapour handling capacities of an oil-sealed pump or prevent cavitation in liquid ring pumps.

6.5.2 PUMP SIZING FOR VESSEL EVACUATION (WITHOUT ANY PROCESS GASES OR VAPOURS)

The pumpdown time is calculated using the below equation, assuming the system is leak free, and the pump selected can achieve a vacuum level at least one decade lower than the required operating pressure, (*i.e.*, $p_u < 0.1 p_T$).

$$t = \frac{V}{S} \cdot \ln \frac{p_a}{p_T} = \frac{V}{S} \cdot \ln \frac{1013}{p_T} \qquad (6\text{-}2)$$

where:

t = time [h, min, s],

V = system volume [m³, l],

S = effective pump speed [m³/h, l/min, l/s],

p_a = atmospheric or initial pressure [1013 mbar(a), etc.], and

p_T = operating pressure [mbar(a), etc.].

Incorporating leak rates, (L in mbar(a)l/s and outgassing rates (G in mbar(a)l/s), complicates the pumpdown calculations, as given in below equation.

$$t = \frac{V}{S} \cdot \ln \frac{(p_a - p_u) - Q/S}{(p_T - p_u) - Q/S} \qquad (6\text{-}3)$$

where:

Q = gas load, $L + G$ [mbar(a)l/s],

V = system volume [m³, l],

S = effective pump speed [m³/h, l/min, l/s],

P_u = ultimate (lowest) pressure of pump [mbar(a)]

p_a = atmospheric or initial pressure [1013 mbar(a), etc.], and

p_T = operating pressure [mbar(a), etc.].

Finally check by calculating the lowest system pressure (p_i) achievable using the below equation, by using consistent units.

$$p_i = \frac{Q}{S}\frac{[\text{mbar s}^{-1}]}{[\text{l s}^{-1}]} \qquad (6\text{-}4)$$

p_i should be lower than p_T.

The time needed to evacuate a chamber can be calculated given the system volume, pump speed, the initial pressure and the required pressure. Edwards can assist in pump down calculations with its in-house calculation software. If air leakage is significant, it needs to be taken into account.

6.5.3 PUMP SIZING TO MAINTAIN PROCESS PRESSURE (WITH FLOW OF GASES OR VAPOURS)

In a typical chemical or pharmaceutical process, the pump down time is not the only critical parameter for the process. It is also important to size the vacuum pump in such way that it will be able to maintain the required pressure during the process, where process gases and vapours are present.

To size the pump system accordingly the total amount of gas, *i.e.* condensable vapours and non-condensable gas (incl. air leakage), needs to be determined. The total volume of gas can be calculated from the Ideal Gas Law:

$$V_i = \frac{\dot{n}_i RT}{p_{tot}} \qquad (6\text{-}5)$$

$$\dot{V}_{tot} = \sum \dot{V}_i \qquad (6\text{-}6)$$

where:

V_i = volume of gas, *i* (condensable and non-condensable arriving at the pump inlet) per time unit (m³/h) $= \frac{dV_i}{dt}$

V_{tot} = total gas volume per time unit (m³/h)

n_i = number of kmoles of gas, *I* ,per time unit $= \frac{\dot{m}_i}{M_i}$

R = universal gas constant

m_i = mass of gas, *i* ,per time unit

M_i = molecular weight of gas, *i*

T = temperature at the pump inlet [K]

p_{tot} = total system pressure at the pump inlet [kPa]

p_{tot}	V	T	R
Pa (Nm^{-2})	m³	K	8.314 J mol^{-1}K^{-1}
mbar(a)	dm³	K	83.14 mbar(a) litres mol^{-1}K^{-1}
dyne cm^{-2}	cm³	K	8.314 x 10^7 ergs mol^{-1}K^{-1}
mmHg (Torr)	dm³	K	62.364 mmHg litres mol^{-1}K^{-1}
atm	cm³	K	82.058 atm cm³ mol^{-1}K^{-1}

Note: 1 mm Hg = 1 Torr
Table 6.1 Values of R (for 1 mol) in Various Systems of Units. Calculated for STP.

When a condenser is used at the pump inlet, the amount of vapours condensing out at the given condenser operating conditions (cooling temperature, pressure) can be subtracted from the total gas load. Edwards can assist in assessing the total amount of condensables saturating the non-condensable gas load using a condenser.

The throughput needed to maintain the required process pressure, can be calculated using equation (2-7):

$$Q = p \cdot \dot{V}_{tot} = p \cdot S_{net}$$

where:

Q = flowrate or throughput [mbar.m³/h]

p = process pressure

V_{tot} = total gas volume per time unit [m³/h]

S_{net} = effective or net pumping speed [m³/h]

Therefore, the required net pumping speed can be obtained by rewriting the equation:

$$S_{net} = \frac{Q}{p}$$

Note, that this gives the net or effective pumping speed for the system. Any losses, due to conductance of inlet piping or pressure drops caused by a condenser need to be taken into account and added to the required pumping speed of the vacuum pump.

6.6 PRESSURE CONTROL

Controlling the pressure during a process run can be essential to assure the quality and yield of the product. There are three methods commonly used to achieve this:

a) Air bleed – A defined amount of air or nitrogen is introduced to the system to maintain a constant pressure.

b) Throttling – A throttling or control valve is placed at the inlet of the pump to control the pressure. This is only possible, if a vacuum pump is used that can run continuously against a closed inlet valve. Most dry pumps will happily do so. In cases where a dry pump is used in combination with a mechanical booster, it may be possible to place a control valve between pump and booster.

c) Variable speed drive – State-of-the-art technology are dry pumps equipped with a variable speed drive. By varying the frequency, the capacity of the pump and hence the pressure of the system can be controlled. For a pump/booster combination, either the dry pump itself or the mechanical booster or both can be run on an inverter.

6.7 HANDLING VAPOURS

When vapours are pumped, working pressure and pumping speed are not the only two factors that need to be considered. A third factor, namely the partial pressure of the vapour, becomes important for determining the vacuum system arrangement. The partial pressure of the vapour may vary considerably during a process. Additionally, condensation inside the swept volume of the vacuum pump should be avoided. This can be done by several different means. This chapter will describe the use of gas ballast and purge gas, as well as the use of condensers to pump vapours and condensables.

6.7.1 USE OF GAS BALLAST & PURGE GAS

Gas ballast (GB) is a constructional feature widely applied in many primary vacuum pumps for the pumping of vapours. Wolfgang Gaede invented the gas-ballast principle in 1935 and was originally focussed on primary oil-sealed pumps. Gas ballast is useful in reducing the extent of vapour contamination in the oil (and/or other parts) thus extending oil life and, at a more fundamental level, permitting the pump to operate on a vapour duty at nearly full specifications. Put simply, depending on the percentage make-up of vapour in the pumped load, then without gas ballast the pump can 'stagnate'.

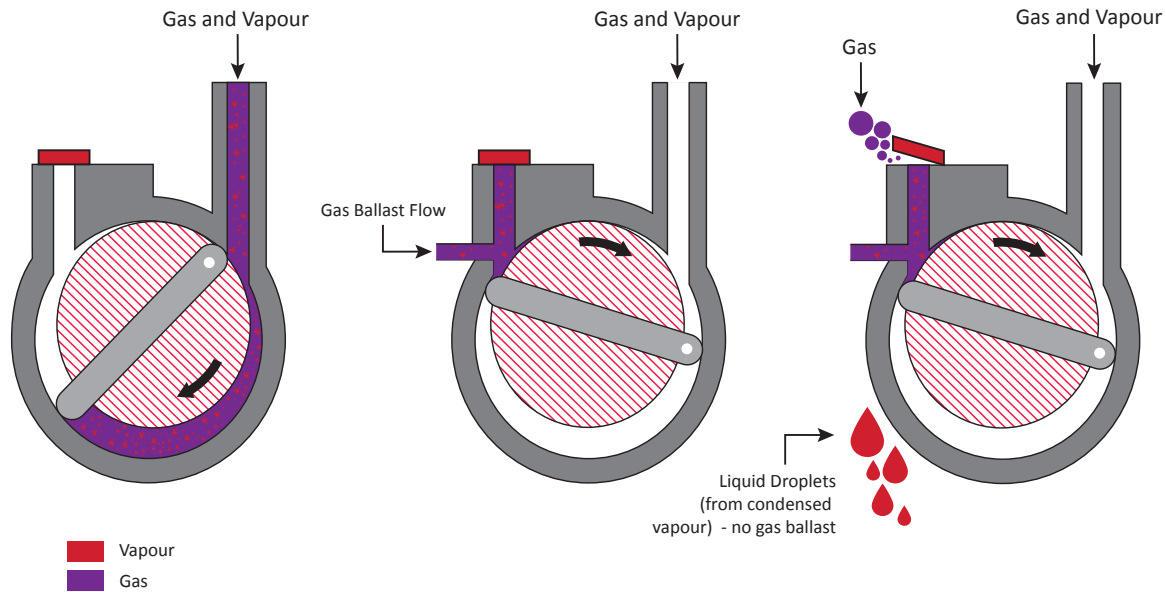A typical physical representation of the gas ballast configuration is shown in Fig. 6.2 below:

Figure 6.2: Gas ballast configuration

The basic principle of ballast is that atmospheric air (or CDA or inert gas) is admitted into a pump during the (late) compression stages (as above). This increases the percentage of non-condensable gas such that the partial pressure of the vapour being pumped is below its saturated vapour pressure when the exhaust valve opens (typically at a pressure of 1.05 to 1.2 bar(a)). This means that the vapour is discharged from the pump without liquefaction.

Note that the term vapour pressure usually refers to the saturated vapour pressure.

The maximum vapour handling capacity (MVHC) of a pump is the maximum amount of vapour that can be pumped without condensation in the pump (as discussed above); the vapour being compressed such that it just avoids saturation at the point of exhaust. The Maximum Water Vapour Handling Capacity (MWVHC) refers to the specific case of water vapour handling and this is a measure quoted for pump performance and is used a relative measure.

Note that the speed of the pump is not a determining factor to the MVHC. However it does influence the degree of degradation of the inlet pressure and the operating pressure at a given vapour flowrate. The operating temperature of the pump, the exhaust valve opening pressure and the gas ballast flowrate are the major factors in maximising the MVHC.

The gas ballast flowrate will be determined by a combination of the considerations of the vapour capacity required and the degrading effect the ballast flow has on the inlet pressure. The positioning of the gas ballast introduction point to the pumping medium (*e.g.* ensuring the correct point in the wraps of a scroll pump) is crucial: It has to be close enough to the pump inlet to ensure no condensation during the progressive compression, but at a point where the ultimate pressure during ballast flow is not overly corrupted.

Comments

- MVHC is independent of pump speed/displacement: the operating temperature of the pump, the exhaust valve opening pressure and the gas ballast flowrate are the major factors in maximising the MVHC.

- Gas ballasting can also be used 'retrospectively' to purge condensed and dissolved vapours from the oil/other materials of the pump's mechanism.

- Generally a pump should be operated with ballast and allowed to reach full temperature (~30-60 mins) before any condensable vapours are introduced. Similarly after vapours have been processed the pump should be operated on GB for another 30-60 mins before turning the pump off (this prevents condensation in a non-running, cooling pump).

- Gas ballast should be introduced at the point where it has least effect on the ultimate pressure, but avoids condensation occurring in compression.

- When using GB an oil-sealed rotary vane pump an oil return kit may be needed as there will be carry over oil loss.

- The effect of ballast humidity is small and is neglected (usually in the case of pumps running at a temperature > 70°C).

Purge gas

In some cases using the gas ballast port is not sufficient to prevent condensation inside the pump; in particular when high loads of vapours are pumped. Then, alternatively or in addition to the GB, air or an inert purge gas can be introduced at the pump inlet. This allows for higher gas flows and hence higher dilution of the vapour. Subsequently, a lower partial pressure of the vapour is achieved.

Note that a higher flow of inlet purge gas will also impact the ultimate pressure of the pump. For oil-sealed pumps a higher gas flow also means a higher carry over oil loss. Therefore, inlet purge is commonly used with mechanical dry pumps.

Applying an exhaust purge is an effective way to prevent condensation at the pump exhaust and the exhaust line.

6.7.2 USE OF A CONDENSER

Chemical and especially pharmaceutical vacuum processes can generate vapour loads of such magnitude that direct pumping by mechanical means would prove extremely expensive and therefore uneconomical. A well-designed vacuum condenser is by far the most economical device to pump large quantities of condensable matter from a vacuum system. Since total condensation cannot be achieved, the condenser must be backed by a vacuum pump to remove the permanent gases and compress them to atmospheric pressure. It should be noted that the gas will be saturated with vapours. Therefore, the backing pump should be adequately sized to cope with the exit load from the condenser.

Most chemical processes such as distillation, drying, etc., are mass transfer devices whereby large quantities of liquids are evaporated in a vessel, such as a distillation column or a dryer, and transferred or pumped away. In the case of distillation, the transferred vapour, when condensed back, represents the main product; thus distillation plants are packaged to include the evaporator and condenser to recover the distillate. The vacuum pumping requirements are then specified based on the permanent gas load or leakage rate of the package, and the entrained or saturated vapour on the exit of the condenser.

In contrast, a dryer will normally be charged with a more or less solid material which represents the product in its next to finished state, requiring unwanted and storage life limiting solvents, mostly water, to be removed. The solvent removal means, generally, vacuum equipment is specified on the duty or capability of the dryer and vacuum pumping will include vacuum condensation, if applicable.

Typically, helical coil condensers or shell & tube condensers are used in combination with mechanical pumps. Fig. 6.3 shows the schematic of a typical shell & tube condenser.
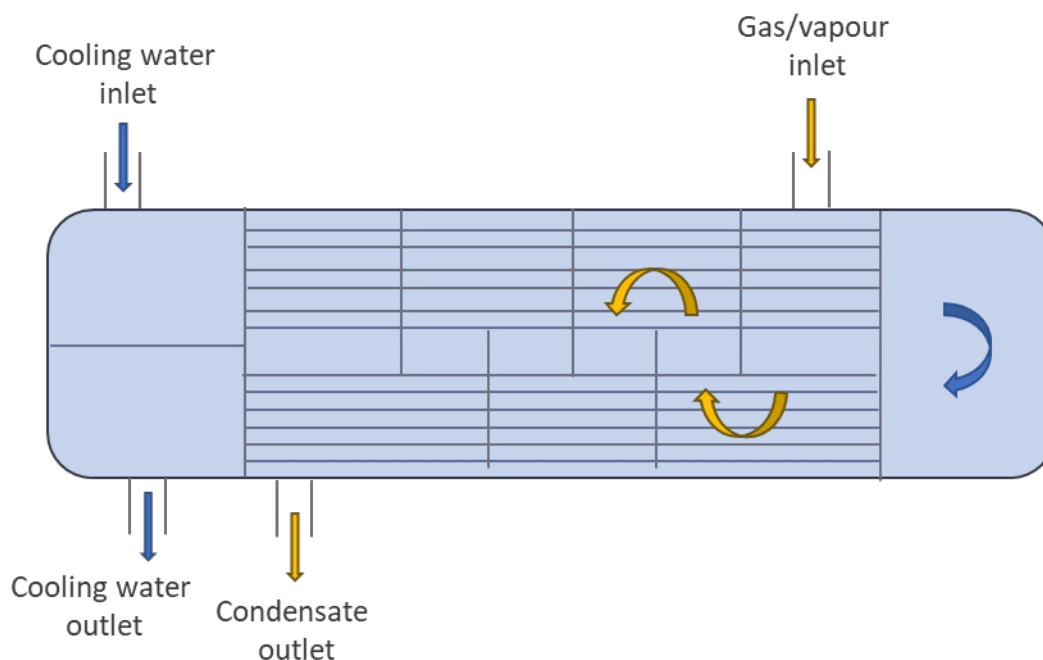

Figure 6.3: Schematic of a shell & tube condenser.

6.8 HANDLING FLAMMABLES & EXPLOSIVES

During a pharmaceutical manufacturing process, it is often required to pump potentially flammable or explosive atmospheres. Therefore, it is necessary to consider the possibility of ignition sources resulting from failure conditions of the equipment. When installing vacuum pumps, a safety strategy needs to be developed that should include the following steps:

1. Identification of explosive atmospheres
2. Avoiding explosive atmospheres
3. Eliminating possible ignition sources
4. Limiting the effects of a potential ignition

The below sections will look into the specific aspects of each of these steps.

6.8.1 IDENTIFICATION OF EXPLOSIVE ATMOSPHERES

Understanding what the explosive atmosphere is and where an ignition might take place is a vital first step. Therefore, the internal atmosphere from the process interface and inside the vacuum pump, as well as the external atmosphere surrounding the pump must be taken into account. The source of explosion hazard is generally an oxidant (*e.g.* O_2, O_3, F_2), a flammable or explosive material (*e.g.* H_2, solvent vapours, fine metal dust) or pyrophoric materials (*e.g.* silane, phosphine).

6.8.2 AVOIDING EXPLOSIVE ATMOSPHERES

A flammable material will only create a potentially explosive atmosphere, if it is combined with air, oxygen or other oxidants and its concentration lies between the Lower Flammability Limit - LFL (or Lower Explosion Limit - LEL) and the Upper Flammability Limit - UFL (or Upper Explosion Limit - UEL). The explosion hazard can be avoided by ensuring that the concentration of the potentially flammable mixture is kept outside the flammable zone. Hence, an explosion will not occur even with an ignition source present. There are a number of strategies that can be used to avoid operating with gas mixtures in the flammable zone. The choice of strategy will depend on the outcome of the risk assessment (hazard analysis) for the process and the pumping system:

a) Maintain the flammable gas concentration below the LFL (LEL)

To minimize the risk of the flammable gas accidentally entering the flammable zone, a safety margin for below-LFL (LEL) operation should be used. A safety margin should be determined following a risk assessment. Some authorities suggest maintaining the concentration at below 25% LFL (LEL). The commonly used method of maintaining a suitable concentration below LFL (LEL) is dilution with an inert gas purge (*e.g.* nitrogen), introduced into the pump inlet and/or purge connections. The required integrity of the dilution system and of any alarms or interlocks will depend on the hazardous zone which would result if the dilution system were to fail.

b) Maintain the oxygen concentration below the MOC (LOC)

This mode of operation requires the use of oxygen concentration monitoring of the pumped gases to ensure safe operation. To minimize the risk of the flammable gas accidentally entering the flammable zone, a safety margin for the Minimum Oxygen Concentration - MOC (or Limiting Oxygen Concentration - LOC) operation should be used. Available industry standards indicate that where the oxygen concentration is continuously monitored, it should be maintained at less than two volume percentage points below the lowest published MOC (LOC) for the gas mixture. Unless the MOC (LOC) is less than 5%, the oxygen concentration must be maintained at no more than 60% of the MOC (LOC). If monitoring is only undertaken in the form of routine oxygen level checks, the oxygen level should not be allowed to exceed 60% of the lowest published MOC (LOC) unless the MOC (LOC) is less than 5%, in which case the oxygen concentration must be maintained below 40% of the MOC (LOC).

The preferred method of maintaining the oxygen level below the lowest published MOC (LOC) is by the rigorous exclusion of air and oxygen from the process and pump system, together with dilution of the pumped gas with an inert purge gas (such as nitrogen), introduced into the pump inlet and/or purge connections, if needed. The required integrity of the air/oxygen exclusion measures and of any alarms and interlocks will depend on the hazardous zone that would result were the exclusion and dilution systems to fail. Precautions typically required to rigorously exclude air from the process and pump system are given at the end of this section.

c) Maintain the flammable gas concentration above the UFL (UEL)

Where flammable gas concentrations are high, then operation above UFL (UEL) can be more suitable. To minimize the risk of any accidental incursion into the flammable zone, a safety margin for above UFL (UEL) operation should be used. It is recommended that the residual oxygen level in the gas should be maintained at less than 60% of the absolute oxygen level normally present at the flammable gas UFL (UEL) concentration.

The preferred method of maintaining the oxygen level below this safety margin, is rigorous exclusion of air and oxygen from the process and pump system. Dilution of the pumped gas with an inert purge gas (such as nitrogen) or with additional flammable gas ('padding' gas), introduced into the pump inlet and/or purge connections, may also be needed. The required integrity of the air exclusion measures, of any purge gas introduction system, and of any alarms and interlocks will depend on the hazardous zone that would result were the exclusion and dilution systems to fail.

d) Maintaining the flammable gas concentration below the minimum explosion pressure

Every flammable material has got a minimum pressure below which an explosion can't be sustained. If the pressure at the inlet of the vacuum pump can be maintained securely below this pressure then ignitions starting inside the vacuum pump will not be able to spread. Precautions, however, must be taken for the exhaust of the vacuum pump. Edwards research has proven that it is not possible to sustain a burn in a potentially explosive atmosphere below 60 mbar(a).

Precautions typically required to rigorously exclude air from the process and pump system are as follows:

Elimination of air leaks

Use a leak detector or conduct a pressure rise test (refer to section 5.2). Before admitting flammable materials into the process chamber, it is possible to perform a test to establish that air (oxygen) leakage into the vacuum system is within allowable limits.

To perform a pressure rise test, the empty process chamber is evacuated to a pressure just below the normal operating pressure, and is then isolated from the vacuum pump. The pressure in the process chamber is then recorded over a fixed period of time. As the volume of the process chamber is known along with the maximum allowable air leakage, it is possible to calculate a maximum allowable pressure rise that can occur over the fixed period of time. If this maximum pressure limit is exceeded, action must be taken to seal the source of the air (oxygen) leakage into the process chamber; the test must then be repeated successfully before the admission of flammable materials into the process chamber is allowed. In some cases, the ability of the vacuum system to achieve a good base pressure can be used to indicate system leak tightness.

Remove all air from the system before the start of the process

Before any flammable gas is admitted into the process, the system should be fully evacuated and/or purged with inert gas (such as nitrogen), to remove all air from the system. At the end of the process, this procedure should be repeated to remove any flammable gas before the system is finally vented to air.

Reverse flow

The system operating procedures and facilities need to protect the system from any reverse air flow, which might result from a pump failure. Any pumped flammable gases need to be safely disposed of at the final vent from the pump exhaust. Flammable gas mixtures should not be able to arise in the exhaust pipeline. This can be achieved by the use of suitable inert purging of the pipeline before the start of and after the end of the flammable gas process, and by the use of adequate inert gas purging.

6.8.3 ELIMINATING POSSIBLE IGNITION SOURCES

Where vacuum pumps are used to pump flammable mixtures, all possible sources of ignition must be considered. Below are some areas of consideration, which can be used as part of an overall review. Depending upon the process it might be possible to avoid some or all ignition sources. If it is not possible to avoid the ignition source because of process conditions or system requirement, the system needs to be designed accordingly.

Mechanical contact - Mechanical contact of rotating and stationary parts inside the vacuum pump and system could provide an ignition source. All Edwards vacuum pumps are designed and built to keep the correct running clearances inside the pump during all operating conditions. To avoid this ignition source it is important to avoid deposition of materials on the internal surfaces or to clean the pump. The bearings must be kept in good condition, have sufficient lubrication and suitable purge gas to eliminate contact with process gases. The recommended maintenance regime for the bearings must be followed to ensure safe and reliable operation.

Particle ingestion - All pumping mechanisms have the potential to ingest particles which have been created by the process or which are a result of the system manufacturing process. Where these are rolled between a moving surface and a static one, it is possible to generate heat. A suitable inlet screen (mesh) or filter will prevent the ingress of particles into the vacuum pump to reduce the size and volume of particles to a safe amount. Care must be taken to have a suitable maintenance regime for the inlet screen.

Dust build-up - The build-up of fine compacted dust within internal clearances can occur where any pumping mechanism is placed on a dust generating process. Even with the use of inlet dust filters, it is still possible for small dust particles to enter the pump. With small dimensional changes due to thermal changes, compacted dust can touch a moving surface and create heat.

Heat of compression (auto-ignition) - The internal heat of compression within any compressor must be considered in relation to the auto-ignition temperature of any gases or vapours which are pumped. It must be ensured that the pump has a temperature classification that is at least the same or higher than the gases being pumped.

Hot surfaces - Where flammable gases or vapours are allowed to come into contact with a hot surface, they may ignite if the auto-ignition temperature is exceeded. Note: Edwards pumps and flame arrestors should not be thermally insulated if this could cause increased surface temperatures internally (and externally) leading to auto-ignition.

Externally applied heat - Externally applied heat can occur, for example, in the event of a fire in the immediate area of the vacuum equipment. Under this condition, it is possible to generate internal pressures in excess of the maximum static pressure of the system, and temperatures in excess of auto-ignition temperature. This should be considered as part of the system hazard analysis.

Hot process gas flow - High inlet gas temperatures can lead to internal (or external) surfaces exceeding the auto-ignition temperature of the materials being pumped. High temperature inlet gas can also lead to rotor/stator seizure. For maximum allowable internal gas temperatures, the vacuum pump instruction manual should be consulted.

Catalytic reaction - The presence of certain materials can lead to catalytic ignition. All materials of construction in the vacuum system should be considered for their potential to act in this way with the pumped gases or vapours.

Pyrophoric reaction - The heat of combustion of pyrophoric materials caused by air or oxidant ingress could act as an ignition source for any flammable material present.

Static electricity - Certain conditions can occur where static electricity can build up on insulated components before discharging to earth in the form of a spark. The potential for static build-up should be considered as part of the system design.

Lightning - Where located in an outdoor location, a lightning strike can provide ignition energy. The potential of this event occurring should be considered as part of the system design.

6.8.4 LIMITING THE EFFECTS OF A POTENTIAL IGNITION

Where it is not possible to avoid the flammable zone, the equipment needs to be designed to avoid or to contain any resulting explosion without rupturing or transmitting a flame to the outside atmosphere. If the external atmosphere of the vacuum system is hazardous, all equipment must be suitably rated for it.

Within the European Union the ATEX directive gives clear guidance on the design of equipment that is to be used in potentially explosive atmospheres.

a) ATEX directive

ATEX is an amalgamation of the French words 'Atmospheres' and 'Explosibles' which means 'Explosive Atmospheres' and was realised with the introduction of the ATEX directive 94/9/EC in 2003 and recently updated with the new directive 2014/34/EU in 2016. This European Union (EU) legislation standardises the way to use any equipment (mechanical or electrical) in potentially explosive atmospheres safely; to ensure that no harm can come to the people in the vicinity and to the environment.

ATEX is an EU legislation and as such is law and adherence to it is mandatory within the European Union. Before the directive came into force the use of equipment in explosive atmospheres was mandated by national laws and working practices. Most of these are historically evolved from the experience gathered in the mining industry and were mostly limited to electrical equipment.

So how is compliance to the ATEX directive shown?

Depending on the type of equipment (mechanical or electrical) and on the equipment category (category 1, 2 or 3) the ATEX directive dictates that in some cases a notified body

has to certify compliance to the directive whereas in other cases the manufacturer or importer into the EU can self-certify the equipment. Table 6.2 shows which type of equipment can be self-certified by the manufacturer and which ones need the involvement of a notified body.

	Mechanical equipment	Electrical equipment
Category 1	Notified Body	Notified Body
Category 2	Self-Certification	Notified Body
Category 3	Self-Certification	Self-Certification

Table 6.2: Certifiers of ATEX equipment.

Notified bodies are conformity assessment bodies nominated by the European Commission, which is the legislative body within the EU.

In order to aid both the notified bodies and the manufacturers of equipment destined to be used in explosive atmospheres, harmonized standards have been written. Whilst the directive is law and defines the level of safety to be applied, standards give practical guidance to show how to comply with the directive, taking into account state of the art technical solutions. EU standards are written and reviewed on a constant basis by groups of technical experts in each EU member country.

The very basis of every ATEX certification for mechanical equipment is the Ignition Hazard Assessment based on EN80079-36 (EN= European Norm) in which the manufacturer assesses the possible ignition sources based on the likelihood that they become active. Vacuum pumps can be classed as mechanical equipment.

Depending on the equipment category required, measures have to be put in place to ensure that the ignition sources cannot become active during normal operation and during foreseeable or rare malfunctions.

A good example is the overall thermal state of a 'dry,' *i.e.* oil free mechanical vacuum pump. If the temperature of the pump exceeds a certain pump specific value, thermal seizure can occur due to thermal expansion of the rotor. If the rotor expands to a point where it touches the stator, sparks with potentially enough energy to ignite a flammable atmosphere present inside the pump can be created. In order to avoid this happening, the thermal state of the pump needs to be controlled. Several measures, depending on the pump design, can be put in place, for example:

- Using a temperature transmitter to measure the case temperature of the pump,
- Using a temperature transmitter to measure the exhaust gas temperature of the pump,
- Monitor the cooling media supply to the pump.

Each of these measures give one level of protection.

Thermal seizure must not be allowed to occur when the pump is running within the pre-defined operating conditions given in the relevant pump instruction manual. However, during malfunctions like loss of cooling media, there is a risk of too high a thermal load remaining inside the pump leading to expansion of the rotor until the pump seizes. The loss of cooling

media can be considered as an expected malfunction. By mounting a flow switch into the cooling media line, this potential failure mechanism has been prevented.

In order to designate a level of safety to the equipment, different categories have been defined. Depending on the explosive zone present, the correct equipment has to be chosen.

As illustrated in Table 6.3, category 3 equipment will only assure safety during normal operation. However, in the zone it is placed (zone 2) an explosive atmosphere will not occur during normal operation and if it does only for a short period of time. The likelihood of the explosive atmosphere being present at the same time as the malfunction is occurring in the pump is considered by the ATEX directive to be an 'acceptable risk'.

For vacuum pumps the internal zone - defined as all areas inside the pump that can come into contact with the pumped media (gases or vapours) - and the external zone - the atmosphere outside of the vacuum pump - are considered separately.

Area classification		Equipment classification	
Zone (European & IEC Classification)	Definition of Zone	Definition of Category	Equipment Classification
Zone 0	An area in which an explosive mixture is continuously present or present for long periods (most of the time)	Protection assured in the case of two independent failures	Category 1
Zone 1	Explosive atmosphere will be present for some of the time	Protection is assured in the event of foreseeable malfunctions	Category 2
Zone 2	Explosive atmosphere is not expected to be present during normal operation and if so only for short amount of time (for example once a month and then for 30 minutes only)	Protection is assured during normal operation	Category 3

Table 6.3: Classification of ATEX zones and according equipment categories.

Once either the manufacturer or the notified body is assured that all ignition sources cannot become active, taking into account the level of protection required for the equipment category to be achieved, the equipment can be marked as being ATEX certified. All ATEX certified equipment must be marked to make them easily identifiable for use in explosive atmospheres.

The following information can usually be found on the equipment rating label:

- ⟨Ex⟩ All equipment suitable for use in potentially explosive atmosphere must show this symbol,

- Equipment category: I for mining and II for non-mining equipment,
- Explosion protection concept used to prevent ignition sources from igniting a potentially explosive atmosphere,
- If applicable the gas group the equipment can be used in,
- Auto ignition temperature.

A typical marking found on an Edwards ATEX vacuum pump ⟨Ex⟩h IIB T4 Gb is described in Table 6.4.

The ATEX directive is applicable to the EU but increasingly non-EU countries/users are choosing it as a measure to ensure safety in explosive atmospheres. It is to be noted that the end-user is responsible for the correct assessment of their zoning requirements.

⟨Ex⟩	Symbol for an explosion-protected device
h	Conformity to EN80079-36
IIB	Gas group IIB
T4	Gases with an auto ignition temperature of > 135 °C
Gb	Gas and category 2

Table 6.4: Explanation of the ATEX markings.

b) Use of flame arrester protection systems

Flame arresters are not explosion prevention devices. They are designed to prevent the propagation of a flame front along a pipe or duct. A typical inlet flame arrester that can be installed at the pump inlet is shown in Fig. 6.4. Flame arresters offer a large surface area and small conductance gaps to the flame front, and so cause the flame to be quenched. Flame arresters are generally only suitable for use in systems which are used for clean gases or vapours. The explosive energy of gas mixtures increases with pressure. Most flame arresters are designed to protect areas where the internal pressure does not exceed atmospheric pressure. It must be ensured that the operating pressure in the exhaust extraction system leading up to the flame arrester is not allowed to exceed the maximum operating pressure.
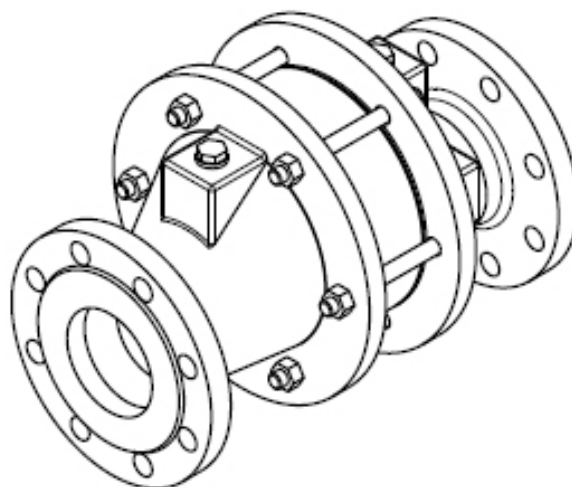

Figure 6.4: Inlet flame arrester (by Protego) to be mounted on a chemical dry pump.

Flame arresters operate by removing the heat of combustion from the flame front, and therefore have a maximum safe operating temperature. The ability of a flame arrester to arrest a flame depends on the speed of the flame front, which in turn depends on its distance from the source of ignition. When used with Edwards chemical vacuum pumps they should be closely coupled to the inlet and exhaust. The use of Elbows and Tee pieces between the pump and the arrester is acceptable for some pumps under certain conditions.

If the mixture of pumped gases and vapours is flammable continuously or for long periods of time (*i.e.* Zone 0 condition) and if there is a risk of an ignition source becoming active during normal operation or foreseeable malfunction, flame arresters should be fitted as required to the primary pump. Third party certification has been obtained for the use of specific flame arresters with Edwards vacuum pumps, demonstrating their ability to prevent flame transmission along the process pipework or into the surrounding atmosphere.

Where the flammable mixture is present for long periods of time an approved and tested temperature transmitter has to be installed on the inlet flame arrester to detect a continuous burn. If a continuous burn is detected the pump needs to be switched off and isolated from the fuel source. In order to protect the flame arrester and pump thermally under rare malfunctions (Zone 0) of the pump, an exhaust temperature transmitter must be installed in the exhaust of the pump. Switch off points are dependent upon pumping systems.

If either temperature transmitter on the inlet or exhaust reach their maximum limit, indicating a fault condition, then suitable actions must be taken. This is application dependent but could include:

• Stopping the supply of fuel - Closing a valve located on the inlet of the vacuum pump will prevent the supply of fuel into the vacuum pump.

• Stopping the source of the ignition - Stopping the vacuum pump by turning off the power to the motor.

• Inerting the area of the burn - The rapid addition of inert gas into the area of burn (typically, but not always located in the exhaust manifold of the pump), will eliminate the flame. Note that it is possible for a flame to re-ignite if the source of ignition is not removed.

6.8.5 HAZARDOUS ATMOSPHERE COMPLIANCE

Edwards can offer a wide range of products for use in and with explosive atmospheres certified for ATEX, relevant American and other applicable global standards. For regional markets, suitable electrical components like valves, and motors for hazardous location to suit the customer requirements can be chosen and combined with the Edwards chemical pump range to achieve compliance with the country's explosion proof legislation.

The pumps used are designed and manufactured to the same high requirements as demanded by the ATEX directive. ATEX certification has been achieved for all of Edwards' chemical range vacuum pumps up to Zone 1 and with a wide range of pumps going up to Zone 0. By using constructional safety as a protection strategy combined, where appropriate, with flame arresters on the inlet and outlet of the pump, Edwards chemical pumps can achieve a very high level of safety (Zone 0) suitable for the most demanding applications. Our chemical dry pumps, liquid ring pumps and EH Boosters are certified up to Zone 0. All other mechanical boosters are certified up to Zone 1.

6.9 APPLICATION EXAMPLES

6.9.1 FREEZE DRYING

Freeze drying (lyophilization) is a dehydration technique where a product is dried under vacuum at low temperature. The water or any other solvent that is contained in the sample is thereby frozen to a solid (ice) and then removed by turning the ice into vapour. Conducting this process under vacuum allows the solvent to be evaporated without having to pass through the liquid phase. The major advantage of freeze drying is that thermo-labile components are preserved and the original size and shape of the sample is maintained. This is only possible by keeping the material in a frozen state at low temperatures during the entire drying process.

The absence of solvent in the final dried product minimizes the effects of oxidation and other degradation processes, thus allowing it to be stored over long periods without the risk of infections by micro-organisms or compositional changes (genetically or enzymatically).

The steps required to lyophilize (freeze-dry) a product in a batch type process are as follows:

1. Pre-treatment / formulation.
2. Freezing at atmospheric pressure (thermal treatment).
3. Primary drying under vacuum (sublimation).
4. Secondary drying under vacuum (desorption).
5. Backfilling & stoppering.

Typical pre-freezing temperatures are in the range of -40 to -60 °C. During primary drying, the frozen liquid (ice) is removed by the process of sublimation (directly turned into vapour). Primary drying is typically conducted at a pressure of 1 to 0.01 mbar(a) depending on the characteristics of the respective product. For an effective freeze drying process the temperature of the product must be higher than the temperature of the condenser. The temperature difference creates a pressure differential, which forces the water vapour molecules out of the material and towards the condenser.

The secondary drying process has begun, when the product reaches a temperature above its eutectic point. During this step the remaining liquid is desorbed. The removal is controlled and optimized by decreasing the pressure down to about 10^{-3} mbar(a), while increasing the shelf temperature to its allowed maximum, just before denaturation of the product occurs. In order to optimize drying cycle times, the end of the primary and secondary drying step is usually determined by conducting a pressure rise test.

Plant and systems

A typical freeze drying set-up (see Fig. 6.5) consists of four basic components: product chamber, refrigeration system, condenser and vacuum pump, whereas the vacuum pump is pumping the chamber through the condenser to protect it from moisture.

The pressure during the freeze drying process is measured by a pressure gauge; usually a pirani or capacitance manometer. Accuracy and repeatability of the pressure measurements are essential for the control of the freeze drying process.
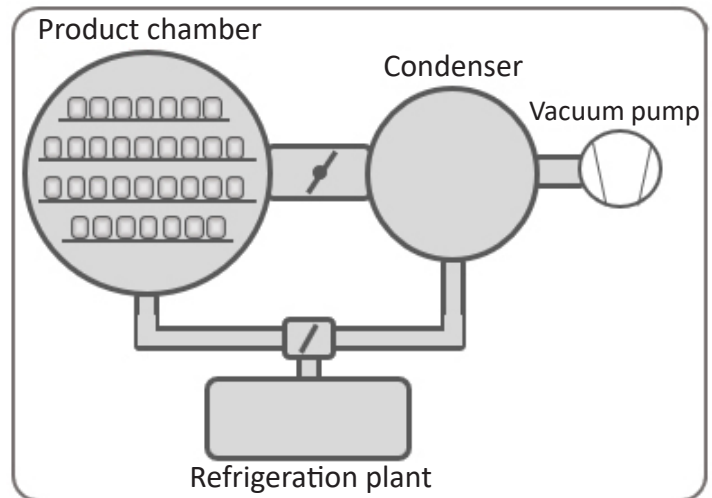

Figure 6.5: Schematic of a freeze drying set-up.

Vacuum pumping

Together with the refrigeration system, the vacuum system forms the heart of a freeze dryer; its main purpose being to evacuate the system down to the processing working pressure. Furthermore, the vacuum system removes all non-condensable gases during the drying process. Pump sizing for shelve freeze dryers can be related back to the actual shelve area, thus a pump capacity of 50 m³/h per 1 m² will give acceptable performance. For general laboratory dryers again 50 m³/h per 10 l of product should be satisfactory. For dryer applications where an externally frozen product is handled, rapid chamber evacuation becomes necessary to avoid excessive temperature rises or thawing. The pump size must than be selected for the required chamber pump down time.

Usually two-stage oil-sealed rotary vane pumps have been used for laboratory and pilot plants. For the larger industrial plants mechanical booster/rotary pump combinations are common. For certain units with exceptionally low condensation temperature, such as liquid nitrogen, vapour booster/diffusion pump backed by a roots/rotary combination have found a place.

As more and more modern pharmaceuticals contain solvents and acids in their formulation it is now quite common to fit dry pump combinations to new and existing drying facilities. This overcomes contamination with hydrocarbons from oil-sealed pumps.

Common pump issues

In the past, two-stage oil-sealed rotary vane pumps have been used and - provided a low temperature condenser was used and only water vapour was being pumped - the pump stood up quite well over time. Frequent oil changes are needed to overcome accumulative contamination and therefore loss in performance. Gas-ballasting provides limited protection.

Lately formulations to be freeze dried contain various solvents other than water. Ethanol, methanol and acidic acid are the most common solvents. These will thin and degrade the pump oil, thus increasing the risk of pump seizure unless the oil is carefully monitored and change frequently. In those cases dry pumps are the accepted way forward as they offer several advantages over oil-sealed solutions. Figure 6.6 shows a typical dry pump combination used for pharmaceutical freeze drying.

Pump sizing information needed

- Material processed,
- Vapours other than water to be condensed/removed,
- Solvents (corrosive / flammable),
- Capacity of dryer in litres or square-meters,
- Plant volume (incl. condenser and pipe work),
- Leak rate if known,
- Evacuation time,
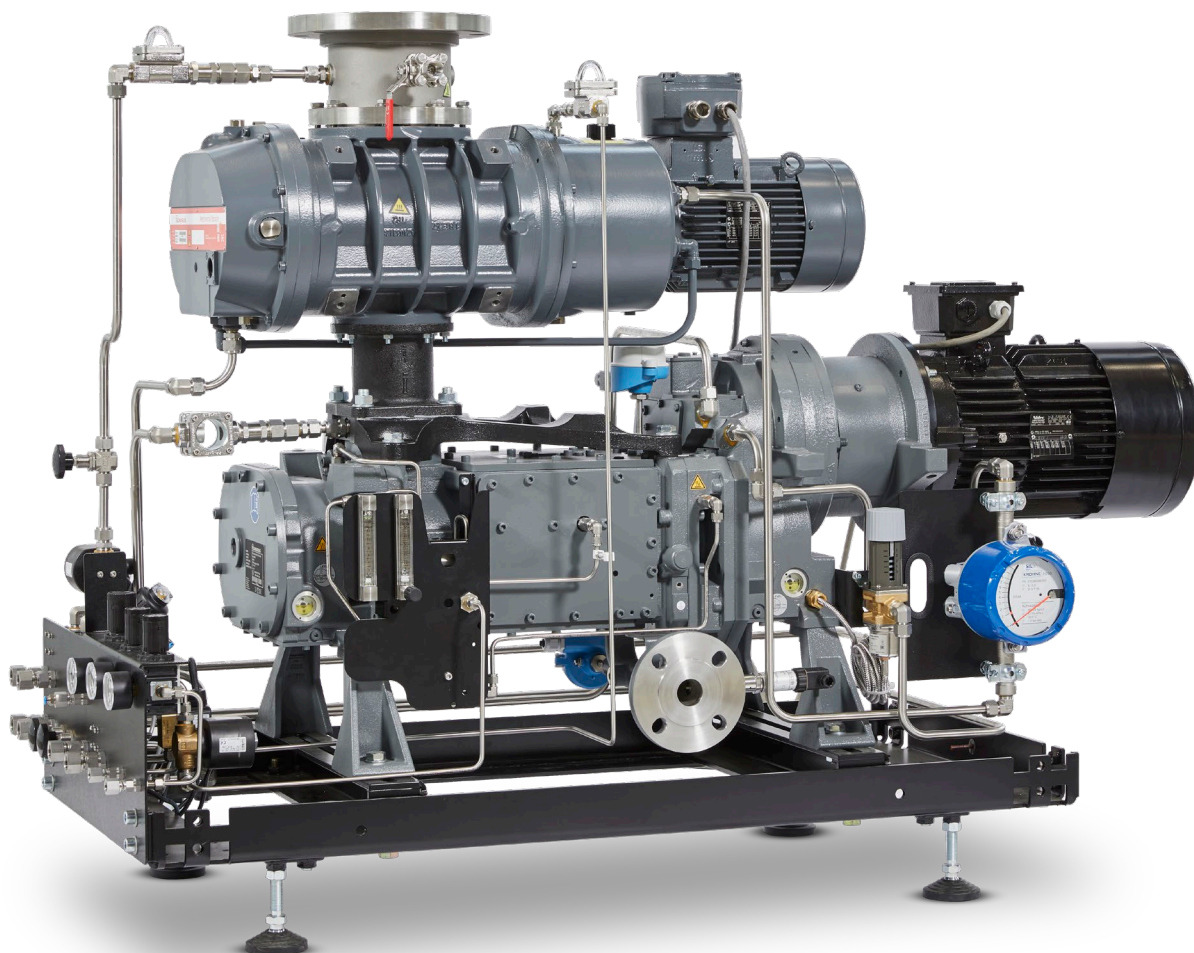- Condenser temperature (max load condition).

Figure 6.6: Combination of an Edwards EDS chemical dry screw pump (bottom) with an EH booster (top), typically used for freeze drying applications.

6.9.2 BULK FILTER DRYING

Vacuum filter dryers are commonly used as:

1. Physical separation method for pharmaceutical products
2. Combination of filtration with vacuum drying
3. Conservation of drying gas by recirculation

Products treated by this method include production of pharmaceutical products and intermediates, fine chemicals, dyes and pigments, plastics and ceramics.

On Nutsche filter dryer applications, the cake is the desired product and the incentive is to obtain a high degree of cake purity by washing, blowing and sometimes by mechanical removal of residual liquids. If washing needs to be more effective, an agitator can be used to re-slurry the cake to allow adequate diffusion of the solvent through the solids. An agitator is also useful in improving the drying rate, the particle size and the consistency of the finished product during the subsequent drying process.

For chemical products, and major intermediates, solute removal during pre-filtration and/or washing is carried out by means of compressed nitrogen. The spent nitrogen is discharged via an incinerator or a vent condenser to atmosphere as waste. Due to current nitrogen costs and stricter regulations on the discharge of contaminated vent gases, recycling of nitrogen is becoming more and more attractive. Final product drying is often done under vacuum. Final drying under vacuum reduces the drying time, achieves lower final moisture or solvent content, occurs at lower temperatures, especially for heat sensitive products, and reduces energy costs. Pumping systems commonly use separate pump sets to produce high vacuum (below 50 mbar(a)) for final drying, and for high-pressure compression for recirculating the nitrogen. A single combined unit compared to separate pump sets can offer significant savings in capital and operating costs.

Pumps and pumping system

As standard drying system, historically liquid ring pump systems are often used (*e.g.* as in Fig. 6.7). To overcome the limited vacuum range of the liquid ring pumps mechanical dry pumps can be used alternatively. One advantage of the dry pumps would be that a drying pressure below 50 mbar(a) can be reached with a single-stage system.

Solvent removal and recovery can be achieved in the liquid ring of the liquid ring pump or at the exhaust of the dry pump using a stand-alone heat exchanger. For filtration and/or nitrogen recirculation, the same liquid ring pump system may be used which is employed for primary drying. For final drying, a separate high vacuum pump set would need to be fitted, providing the required ultimate vacuum to ensure the desired product dryness. Such high vacuum pump sets typically comprise single-stage oil-sealed rotary vane pumps, once-through oil pumps or dry pumps or a combination of mechanical roots blowers backed by a primary pump.
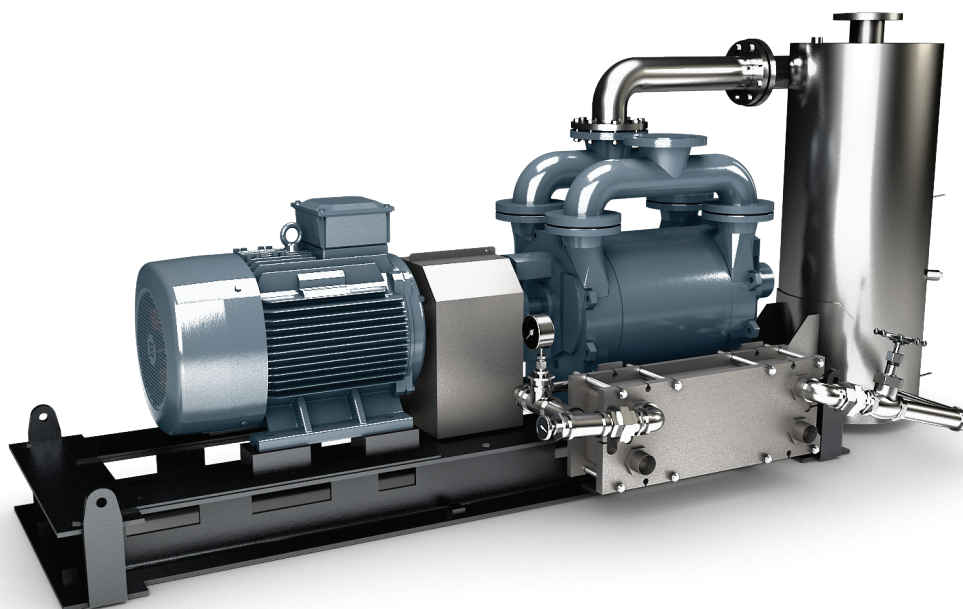

Figure 6.7: Example of an Edwards liquid ring pump with total recirculation system.

Common pump issues

Liquid ring pump capacity and ultimate pressure are directly related to the physical characteristics of the seal fluid properties. As most pumped organic vapours have low boiling points compared to water, considerably lower pressures, *i.e.* <50 mbar(a), can be achieved only if one or more compression stages, such as ejectors or roots blowers, are fitted additionally. As nitrogen consumption and undesirable emissions are the main issues, ejectors running on nitrogen are not considered.

Both dry pumps and liquid ring pumps are susceptible to solids carryover and most filter-drier systems incorporate filtration equipment.

Dry pumps run with higher internal temperatures than liquid ring pumps and thus can suffer from heat-sensitive product thermally degrading or reacting within the swept volume. This may lead to build up of harmful deposits that result in pump seizure.

Pump sizing information needed

- Type of filter-drier and details of the application / process,
- Gas composition,
- Gas flowrate,
- Inlet pressure,
- Exhaust pressure.

6.9.3 CONCENTRATION, EVAPORATION AND CRYSTALLIZATION

Concentration, evaporation and crystallization processes are used to remove unwanted or contaminated fluids. The use of vacuum accelerates the process at lower process temperatures.

Concentration by definition is the process of increasing the strength of a solution and especially the amount of dissolved substance in a given volume of solvent. A product discharged from an evaporator is therefore a concentrated solution, while the product discharged from a crystalliser is a slurry of crystals in a saturated solution. Evaporation leads to an over-saturation and/or sub-cooling, which results in the precipitation of crystals. Evaporation has many similarities with distillation and some share common process vessels. Evaporation and crystallisation as discussed here is a concentration process in that the evaporant is of secondary importance.

Evaporation is the removal of solvents from a solution by vaporisation, which will occur when the partial pressure of the solvent above the liquid level or film is less than the vapour pressure of the liquid. Heating of the liquid increases the vapour pressure and therefore the evaporation rate. Reducing the partial pressure by pulling a vacuum on the liquid also increases evaporation. Applying both heat and vacuum achieves the best possible evaporation results. Heat is always required to raise the feed temperature to its boiling point, to provide the thermodynamic energy for solvent separation and to supply the heat of vaporisation. This process is therefore based an straight forward heat and mass transfer consideration and influenced strongly by economics, particularly operating costs and energy efficiency.

Lowering the boiling point of a liquid by reducing the operating pressure above the liquid is for certain thermo-labile pharmaceuticals a necessity, but can also bring considerable energy savings for higher boiling mixtures. Selecting the correct vacuum levels can bring

the operating temperature down to a level where waste heat source is usable, which may have been lost otherwise. This fact is the main reason why most processes are operated at reduced pressures.

Crystallisation is an important industrial process and many end products are marketed in the crystalline form. As a separation technique, crystallisation is more energy efficient than distillation while yielding a product that has good flow characteristics and is easy to store, handle and pack.

The basic tool to analyse crystallisation is the Ostwald–Miers diagram for solute/solvent (refer to Fig. 6.8). The solubility curve for a system does not depend on process variables. The super-solubility curve however, does depend on process variables and represents the maximum super-saturation a system can tolerate. The super-solubility curve and ultimately the crystal size distribution is affected by the cooling rate and a host of other parameters.
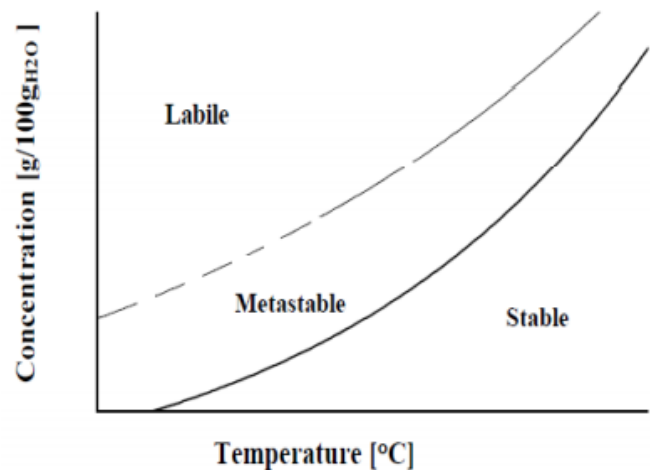

Figure 6.8: Ostwald-Miers diagram for solute/solvent mixtures.

If the solubility of a solid increases with temperature, super-saturation can be created by evaporation, cooling or both. If the curve is relatively flat evaporation is favoured; if it is steep cooling is favoured due to the inherent economics. The end product of a crystallisation process is usually extracted as a slurry of crystals in saturated solution which is fed to a centrifuge for crystal separation. The saturated solution is returned to the process. Recovered crystals may undergo a vacuum drying process to improve flow characteristics.

Plant and system

Evaporator designs are normally a compromise between capital costs of heat transfer areas and energy cost savings. Such compromises have produced numerous types, sizes and configurations of evaporators. Multi-effect evaporators using vapours produced in one effect to heat another effect have low energy consumption but correspondingly high capital costs. The same applies to thermo-compression evaporators where vapours are compressed to condense at high enough temperatures to permit their use as a heating medium in the same evaporator.

Vacuum pumping

Vacuum producing equipment is required to evacuate to and maintain the operating pressure, ranging from 100–0.1 mbar(a). Theoretically, only non-condensables, *i.e.* gases dissolved in the liquid feed, and/or present a result of decomposition as well as leakage air are being pumped. The evaporated vapours are always condensed out for recovery and recycling. As the facility's condenser is usually operated under more or less saturated vapour conditions the vacuum equipment capacity must be able not only to extract the total load of non-condensables but also their saturation with the vapours

at the outlet temperature of the condenser. Crystallisers operating with a final evaporative temperature of the condenser. Crystallisers operating with a final evaporative cooling cycle impose an additional vapour load.

Because of the numerous variables and specific process requirements and conditions individual vacuum pump sizing becomes a necessity and any quoted guide values are meaningless. Steam ejectors and liquid ring pumps or a combination of both are being used, especially for very large installation as found in the main stream production of sugar, sodium chloride, etc. A typical steam ejector/condenser package is shown in Fig. 6.9. Roots blowers have found their way into the vapour recompression cycle especially for multi-effect evaporators where considerable energy savings are possible.

Medium size concentration processes have been served well by liquid ring pumps and are especially applicable where condensate can be used as sealing fluid. With the addition of either air ejector or mechanical booster their useful operating pressure rage can be extended.

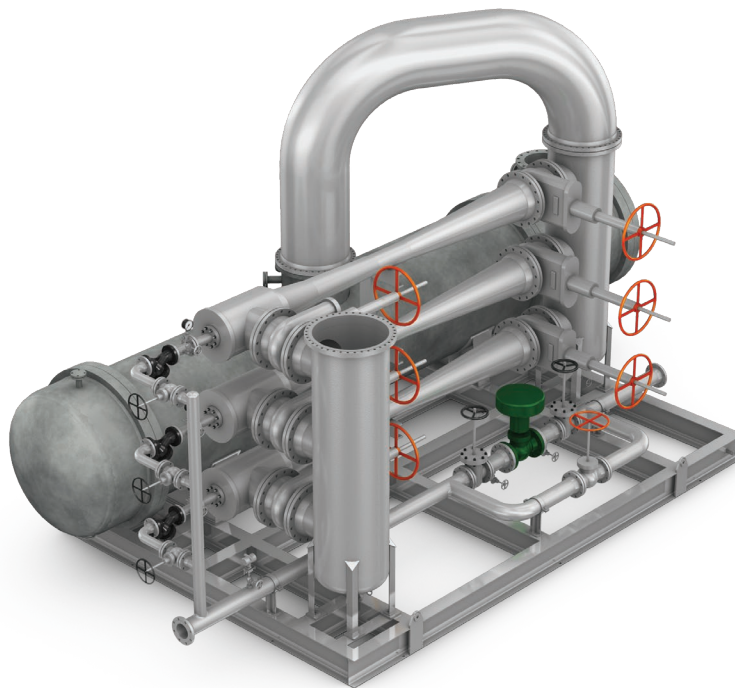Dry pumping is used in some niche applications in the bio-technical and pharmaceutical industry.

Figure 6.9: Edwards steam ejector condenser package.

Common pump types

Usually all pumped loads will contain varying amounts of vapours depending on the efficiency of the condenser and its operating temperature in relation to the present vapour pressure. Unless extensive precautions are taken to prevent vapours from reaching the oil-sealed high vacuum equipment, contamination is most likely to occur and such pump application will have to be seriously scrutinised to avoid major risks. Short, condensation free, falling away pipe connections with or without knock-out pot will alleviate major problems. For rough general application liquid ring pumps stood up well over many years. Dry pumps have proved themselves especially for bio-technical

and pharmaceutical evaporation and crystallisation processes and particularly those with environmental and pollutant problems. In most cases an inlet catch pot with scavenging line must be incorporated to prevent hydraulic lock-up of some dry pumps.

A combination of mechanical booster/liquid ring or dry pump combination should provide the ideal solution for low temperature concentration applications and especially multi-effect systems where the heat of compression produced by the mechanical blower and the dry pump is re-used for product heating.

Pump sizing information needed

- Operating pressure of all process phases,
- Vapour/gas loads/mass flowrates to be pumped from each process phase,
- Physical data of process materials, especially vapour pressures,
- Operating/vapour temperatures of condenser,
- Lowest usable coolant temperature,
- Plant leak rate,
- Type of process/plant, P&ID,
- Pump protection devices available within the plant.

6.9.4 SOLVENT RECOVERY

Regulations and economics increasingly govern the use and emissions of process chemicals, especially volatile organics and pollutants. Such chemicals include alcohols, acetates, amines, esters, phenols, aromatic hydrocarbons and petrol fumes. There are many factors which govern the economics of solvent capture and reuse. These include treatment costs, fresh solvent costs and emissions limits. Solvent recovery processes include absorption, adsorption, desorption, distillation and semi-permeable membrane processes, such as pervaporation. Adsorption, absorption and pervaporation are the main processes using vacuum. Condensation, although quite widely applied, is done at atmospheric pressure. It is cost effective where coolant or cryogenic sources are freely available at relatively no extra cost.

Solvent recovery is split into two distinct applications:
1. Capture and recovery for reuse and
2. Reclaiming and purification for reuse.

Capture and recovery involves the removal and recovery of solvents from process vent gases. Volumetric gas flowrates dictate the technology, size and cost of the solvent recovery system. Solvent concentration in the gas stream is the major influence on the selection, suitability and efficiency of an appropriate system. Reducing the gas flowrate to increase solvent concentration should always be considered to improve the economics of the process, including the vacuum system.

Reclaiming and purification: Solvent recovery or reclamation is often a critical factor in determining the feasibility of a new project or plant expansion. Environmental regulations, new solvent costs and waste disposal costs usually easily exceed the cost of solvent recovery equipment and its operation. Solvent reclamation can be an integral part of the main process stream (such as solvent refining) or accomplished by contractors.

Plant and system

Adsorption/desorption processes

During adsorption, solvent vapour molecules from the air stream make contact with, and are retained on, the surface of a solvent adsorbent where they are physically taken up at 'active sites'. That attraction to the solid surface can lead to condensation of the solvent molecules in the micropores. Solvents that can be recovered using adsorption include acetates, alcohols, esters, halogenated hydrocarbons, ketones, aldehydes, amines, alkanes, aromatics, phenols, oils, carbon dioxide and water.

Adsorption generally releases heat. Desorption requires heat to release the adsorbent of solvents.

Typical adsorbents are highly porous, granular solids with large surface-to-volume ratios. They are sized to provide low resistance to flow. Examples include:

- granular activated carbon (GAC),
- molecular sieves, zeolites,
- macro-porous polymer particles,
- silica gel,
- sodium-aluminium silicates.

The most popular capture systems are based on the twin fixed bed adsorption principle to ensure continuous processing. While one bed is adsorbing the other is desorbing, *i.e.*, regenerated. A typical twin-bed adsorption process is shown in Fig. 6.10.
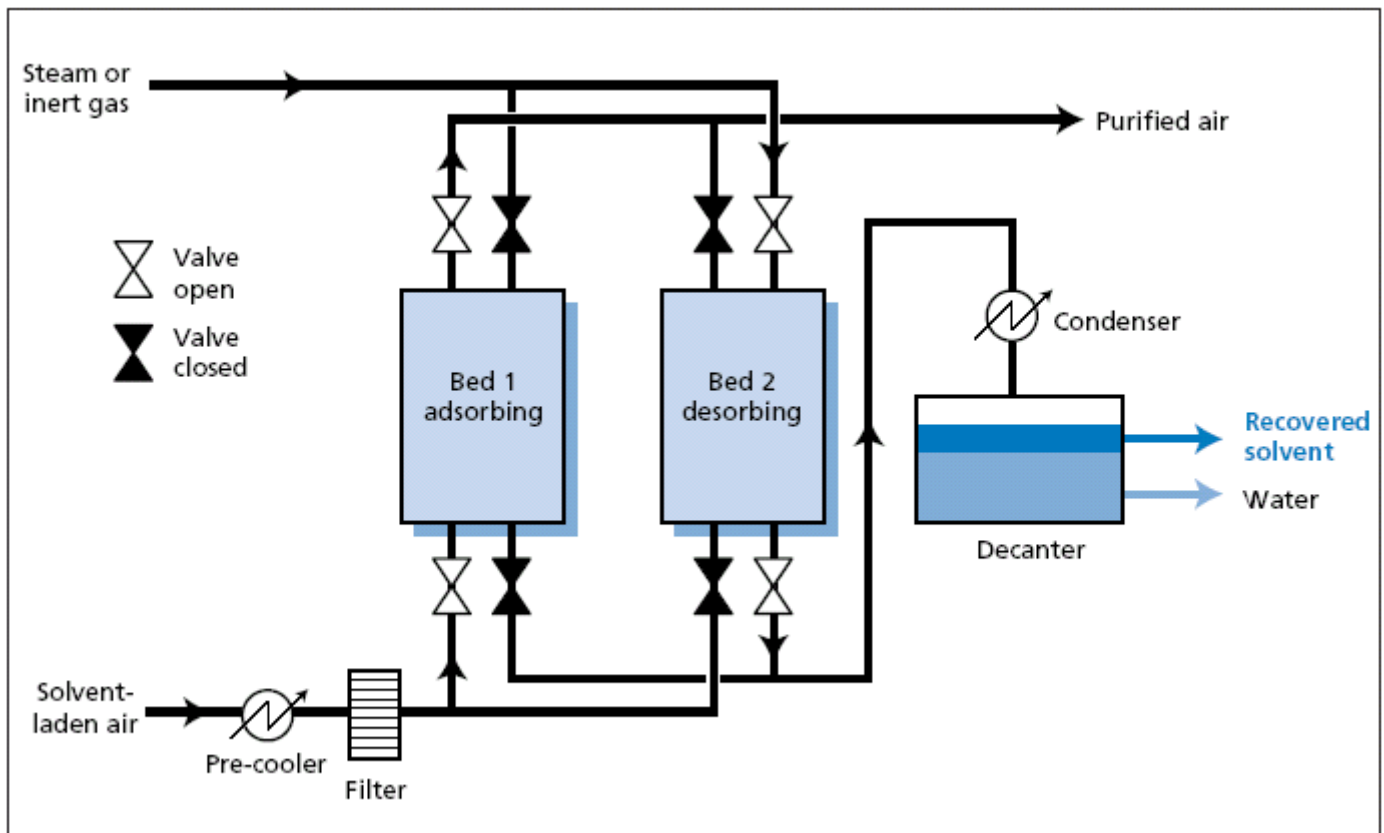

Figure 6.10: Typical twin-bed adsorption/desorption process.

Absorption/desorption processes

Absorption is also a proven technology for solvent capture and recovery for reuse. Absorption, also known as 'scrubbing', involves mass transfer in a gas-liquid contacting device between a solvent vapour and a scrubbing liquid in which it is readily soluble. Physical scrubbing, where the solvent does not react with the scrubbing liquid, is preferred for solvent recovery. Chemical scrubbing, where the solvent reacts with the scrubbing liquid, is used primarily for solvent removal without recovery and is not considered here.

A typical absorption/desorption system comprises two near identical columns. One is for absorption or scrubbing and the other for regeneration or stripping (see Fig. 6.11). The driving force for absorption is the difference between the partial pressure of the solvent in the gas mixture and its vapour pressure in the liquid film in contact with the gas. Absorption does not occur unless this driving force is positive.

Close contact between the gas and the absorbing liquid is essential for effective absorption and can be achieved by breaking up the liquid into small droplets or thin films to provide a greater liquid surface area for mass transfer.
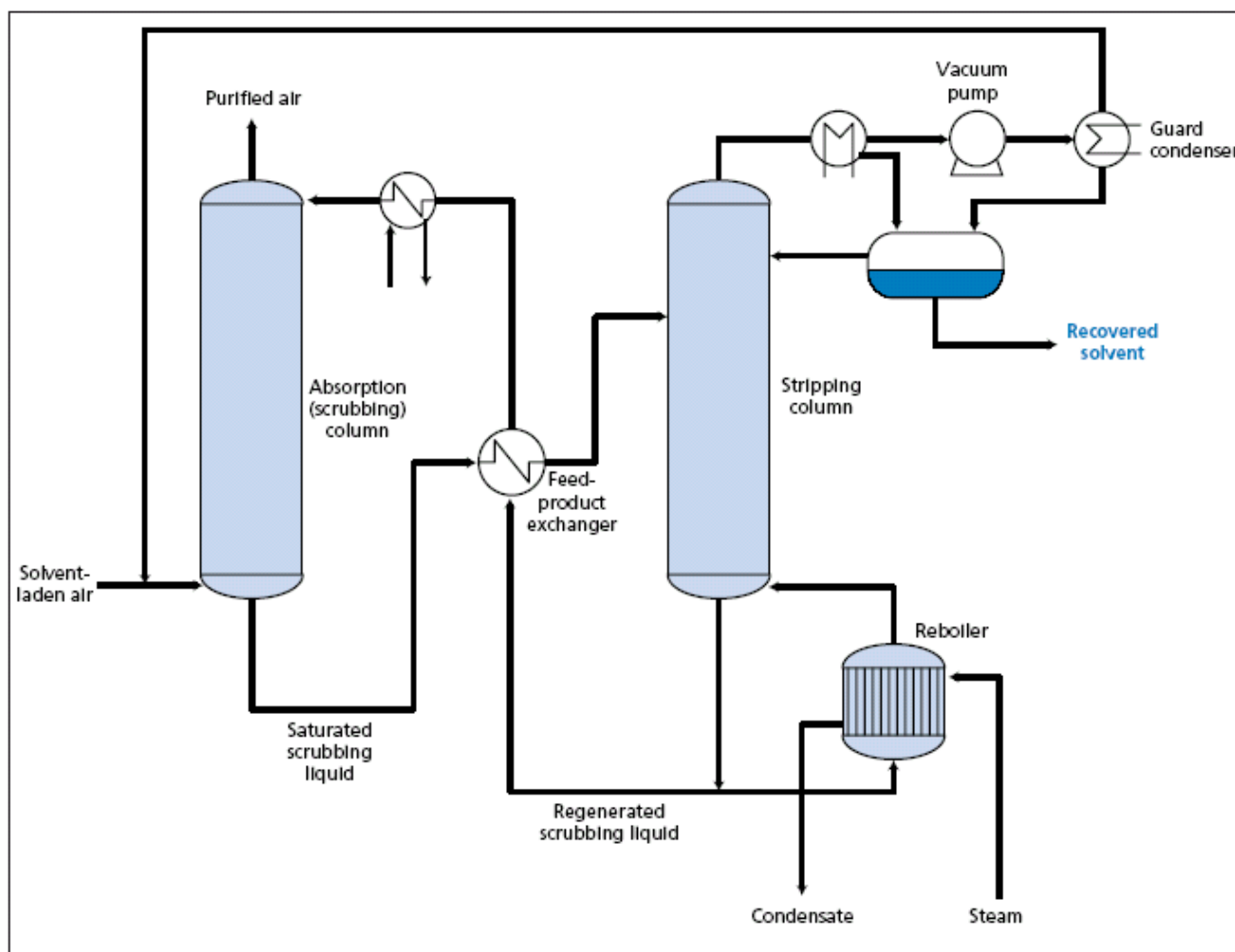

Figure 6.11: Typical absorption/desorption process.

Membrane processes

The use of very thin semi-permeable membranes has come a long way over the last 10-20 years and numerous such solvent capture devices are being installed. Pervaporation is the term used when the feed to the membrane is liquid. If the feed is vapour, the process is called vapour permeation. These processes are typically used to remove water from organics.

The solvent-laden feed stream passes over one side of the membrane and a vacuum is applied on the other side. Driven by its partial pressure the solvent diffuses through the membrane. This results in a more concentrated solvent stream on the vacuum side. The solvents are recovered either upstream or preferably downstream of the vacuum pump. A typical set-up is shown in Fig. 6.12.
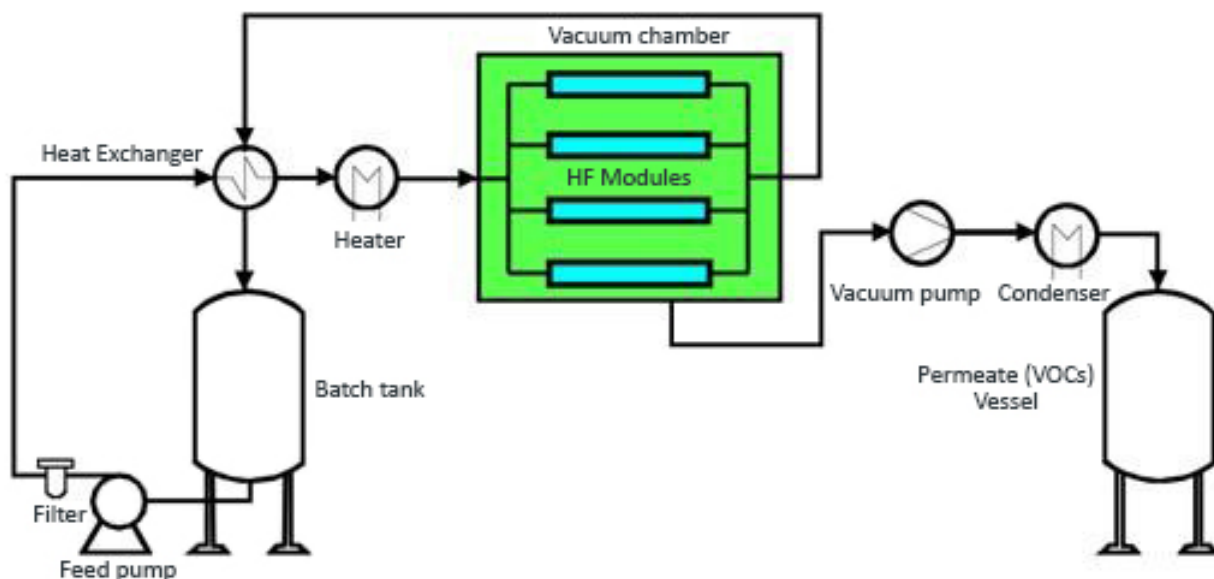

Figure 6.12: Schematic of a parallel multi-module batch pervaporation process.

Efficient separation requires the membrane to be more permeable to the solvent vapour than to air and/or other inert gas molecules. Efficiency depends on the:

- selectivity of the membrane,
- pressure in the air space,
- pressure in the solvent-rich space,
- extent to which solvent vapour has been stripped from the air stream.

If the solvent vapour concentration in the air stream is low, a high vacuum is required on the solvent-rich side of the membrane.

Vacuum pumping

Solvent recovery covers a wide range of processes such as adsorbent and absorbent regeneration through to distillation and pervaporation. Processing capacities can vary from relatively small units of some 10 kg of solvent capture to very large plants coping with several tonnes per hour relating to vacuum pumping speeds of 20 to more than 100,000 m³/h.

For dual column PSA applications a pumping speed of between 500-1,000 m³/h at 50 mbar(a) per tonne of adsorbent for most light solvent will provide a good guide value for regeneration cycles of around 3 hours.

Absorbent regeneration using distillation to recover solvents is based on the mass transfer between evaporator and condenser with the vacuum pump creating the best process pressure for optimum processing conditions. The vacuum equipment will be sized on the mass flow of saturated non-condensables from the condenser. As a guide around 300-500 m³/h at 1-2 mbar(a) per tonnes/h of absorbent circulated is a good approximation. Fuel vapour recovery units require 850–1,250 m³/h at 1013 to 50 mbar(a).

For solvent recovery and purification, the observations above apply for general low value solvents. For the recovery of high value solvents, *e.g.* from essential oil, fragrance and perfume extractions, more sophisticated distillation such as short path distillation is used, requiring larger and more complex vacuum equipment as well. Vapour boosters backed by dry pumps is an economical and environmentally friendly solution, especially if compatible vapour pumping fluids can be found.

Pervaporation vacuum system sizing again is based on mass transfer, similar to distillation. However, operating pressures even for the lighter solvents will be lower, generally at 1 mbar(a) or less. Direct pumping without pre-condensation may require pumping speeds of more than 10,000 m³/h. For optimum recovery rates, exhaust condensation should be used. However, for combination pump sets, interstage condensation can be considered as this will greatly reduce the required backing pump capacity. There is no rough guide values for this process.

Common pump issues

Traditionally liquid ring pumps and multi-stage steam ejectors have been used. Although relatively low in capital costs, these systems come with high operating cost. The condensed steam from the ejectors and the seal fluid from the liquid ring pumps are subject of environmental concerns as this may generate large amounts of waste water. When oil-sealed pumps and pump/booster combinations are used, solvent contamination with oil needs to be avoided. Adequate accessories need to be installed to protect the pump from condensate to ensure reliability and long up-time. The best available environmental option are dry pumps or pump/booster combinations *e.g.* as shown in Fig. 6.13. They also benefit from low costs of ownership and are suitable to reliable operate even with high boiling solvents.


Figure 6.13: Edwards EDP chemical dry pump system.

6.9.5 CHEMICAL REACTIONS

Chemical reactions such as the synthesis of compounds in the chemical and petrochemical industries as well as the fragrance and pharmaceutical industries are widely used. Some of the reactions require reduced pressure in order to avoid high process temperatures, to remove reaction products mainly gases and vapours, or to shift the equilibrium to improve performance and yield.

The effect of pressure on reversible gas phase reactions is covered by Le Chatelier' principle, which states that a system in equilibrium will readjust, if possible to relieve any stress on the system. If one considers the following:

$$3H_2 + N_2 \leftrightarrow 2NH_3$$
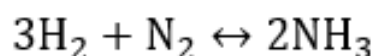
The left to right reaction producing ammonia is accompanied by a reduction in volume from four moles of reactants to two moles of product, resulting in the system pressure decreasing during the reaction. If the pressure is now increased (*i.e.* stress is applied) the chemical equilibrium shifts so as to reduce the pressure by producing more ammonia. Conversely if it was desirable to shift the reaction to the left, the reaction will be operated under reduced pressure *i.e.* vacuum. To maintain the required vacuum condition, and therefore the reaction process, the reaction by-products such as water, hydrogen, alcohols, glycol etc. must be removed continuously. The pressure range for reactions can vary between 50 to 10^{-2} mbar(a).

Plant and system

Apart from the very large commercial reactors (*e.g.* fertiliser production) and cracking plants for hydrocarbons and derivative compounds, which are served by steam ejector + liquid ring pump combinations, most other reactors in the pharmaceutical and fine chemical industry are of smaller capacity of around 2 m³ and below, requiring vacuum pumping speeds of around 100-200 m³/h. Even for the smaller plants steam ejectors and/ or liquid ring pumps were favoured in the past. However with rising energy costs and sharpening environmental regulations, dry pumping is or has been the latest standard.

Reactors are generally vertically mounted cylindrical vessels. Heating jacket and a centrally mounted agitator device are essential features of standard reactors. Although the vessel may be fitted with a removable lid this lid is seldom removed, as all filling, vacuum pumping, temperature and pressure sensors etc. are located there.

Vacuum pumping

Past experience and pilot plant data are usually required to determine the optimum size and operating pressure of a vacuum pump. Data such as reaction rate, reaction order, effect of pressure changes, gas/liquid/vapour ratios etc. need to be considered. The capacity or speed of the pump will have to cope with large quantities of non-condensable reaction gases, including H_2 as well as the usual leakage air associated with such equipment. As the air leakage rate can be very high, especially for older equipment, and as most vapours pumped are flammable, potentially flammable mixtures may be present during the whole or part of the process cycle.

When non-condensable gases in equilibrium with a liquid phase leave the reactor, vapour saturation in addition to any reaction vapour generation must be considered. In most cases the mass transfer data will be known from previous reactor processes and the sizing of the vacuum pump becomes a matter of mass transfer calculations.

Hitherto nearly all types of vacuum producing equipment have been used such as steam ejector + liquid ring pumps using glycol, all types of oil-sealed rotary pumps (pure gas pumping), right up to the more advanced pumps like vapour booster and turbomolecular pumps. The latter pump is of interest for the reaction of organometallic compounds. The type of vacuum pump best suited for any reactor application depends not only on the required operating pressure and capacity, but also on the type of gas and/or vapour reaching the pump. Although numerous protection means, such as condensers, knock-out pots, filters and cyclone separators can provide effective protection, some contamination will penetrate through to the pump.

Figure 6.14: An ATEX certified Edwards EDP/EH chemical dry pumping system with interstage knock-out pot.

Common pump issues

As previously mentioned the choice of the vacuum equipment will depend on the compatibility of all the materials in contact with the reaction gases or vapours.

Oil-sealed mechanical pumps cannot tolerate polymeric or oil soluble materials and acids as these cause further reactions, loss of lubrication, excessive wear, swelling of blades etc. within the pump. Similar considerations apply to once-through oil-lubricated pumps unless very high and uneconomical oil flowrates are set creating possible problems in the disposal of relatively large quantities of waste oil. Steam ejectors and liquid ring pumps are highly reliable, especially when appropriate motive and sealing fluids are selected.

Well engineered dry pumping arrangements provide reliable means for even the most arduous of reactor applications. Well engineered means the incorporation of appropriate protection means as well as safety measures in accordance with the pumped vapours and/ or gases. As mentioned above flammable gas/vapour mixtures will be present during the process cycle, therefore the appropriate correct explosion protection must be provided. An example of an ATEX dry pumping system is shown in Fig. 6.14.

Pump sizing information needed
- Operating pressure of all process phases,
- Vapour/gas loads/mass flowrates to be pumped from each process phase,
- Physical data of process materials, especially vapour pressures,
- Operating/vapour temperature,
- Plant leak rate,
- Type of Process/Plant, P&ID,
- Pump protection devices available within the plant.

7. PRODUCT OFFERING

7.1 DRY PUMP SYSTEMS
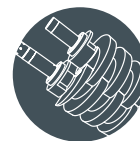
EDP system

Edwards, a world leader in dry vacuum pump technology, successfully pioneered the use of environment-friendly dry vacuum pumps in the early 1980s to meet the highest safety and performance standards. Featuring an award-winning reverse claw mechanism and patented tapered screw technologies, Edwards delivers exceptional performance and energy efficiency with our dry pumps and combinations customised for chemical applications.

Our dry vacuum pumps are capable of handling increasingly complex chemicals, solvents and compounds in a reliable and safe way while ensuring the control and reduction of environmental pollution at a low cost of ownership.

- EDP, EDS, CXS, CDX
- Up to 40,000 m³/h in combination with boosters

- Ultimate vacuum up to 0.001 mbar(a) with boosters

7.2 LIQUID RING PUMP SYSTEMS

LRP system

The Edwards liquid ring pump (LRP) range includes single- and two-stage machines enabling optimum efficiency for processes operating across the process vacuum range. Pump capacities up to 40,000 m³/h are available in single-stage and 7,500 m³/h in two-stage models.

The Edwards LRP range of vacuum pumps is available as standard packages of once through, partial and total re-circulation operation. Liquid ring pumps offer significant advantages when pumping wet gases and vapours or when used for applications requiring special construction materials for corrosion resistance. LRPs can operate with different seal liquids, enabling operations with a liquid compatible with the process.

- HR, LR1A, LR1B, LR1C, LR1D series
- Up to 40,000 m³/h

- Ultimate vacuum up to 0.1 mbar(a) with boosters

7.3 ROTARY VANE PUMP SYSTEMS
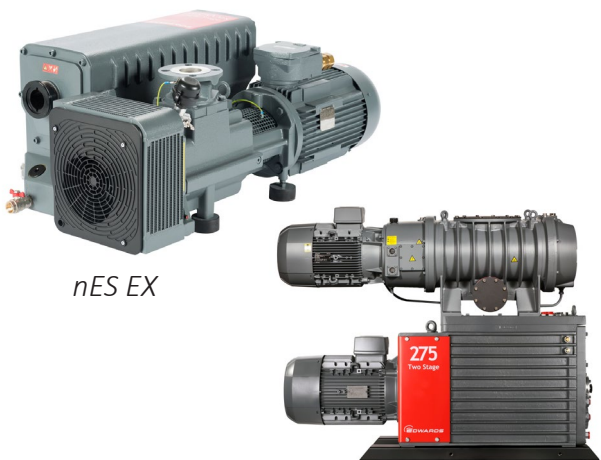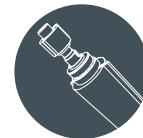
nES EX

E2M/EH Booster Combination

Our Ex series of single-stage and two-stage rotary vane pumps has been specifically designed to handle gases from potentially explosive atmospheres and to operate in environments where a potentially explosive atmosphere can be present. Designed for reliable, long-term operation, our oil-sealed rotary vane pumps are renowned for achieving high ultimate vacuum and rapid pumping speeds, with quiet operation and exceptional reliability.

- nES EX - Up to 3,300 m³/h
- E2M - Up to 2,800 m³/h

- Ultimate vacuum up to 4×10^{-3} mbar(a)
- Ultimate vacuum up to 4×10^{-4} mbar(a)

7.4 VAPOUR BOOSTERS
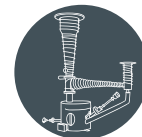
18B4B

30B5M

When higher pumping speeds are needed, our vapour booster pumps are the ideal solution. Field-proven in various industries for more than 60 years, they provide benefits ranging from ease of use, inherent reliability, ease of maintenance and tolerance to a wide variety of inlet and exhaust pressures.

Special variants for applications, such as short path and molecular distillation of fish oils, vegetable oils and vitamins, deodorisation and refining of oils and waxes, and purification of crop protection chemicals, have been created in combination with our vacuum pumps for chemical applications.

- 18B4B, 30B5M
  Capacities up to 15,000 l/s

- Up to 1×10^{-4} mbar(a)
  ultimate pressure

ATEX available on demand.

7.5 MECHANICAL BOOSTERS

EH Boosters

HV Boosters
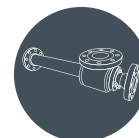

Stokes 6" Boosters

GMB40K Boosters

When coupled with our wide range of vacuum pumps, mechanical boosters increase pumping speed at working pressures, significantly reducing the pump downtime.

- EH boosters, HV boosters
- 250–40,000 m³/h
- GMB40K boosters, Stokes 6" mechanical booster

7.6 EJECTORS

Multi stage ejector system

Steam ejector

Edwards has been instrumental in the development of steam ejectors from the original crude single-stage devices to the highly efficient multi-stage systems currently in use.

Edwards offers single-stage ejector systems and multi-stage systems, providing improved energy efficiency and the capability to handle high vapour loads without the risk of cavitation. They are available in various materials of construction such as carbon steel, stainless steel and a variety of corrosion-resistant alloys with a relatively low capital cost, providing an attractive return on investment.

- Pressure range
  - Single stage: 120 mbar(a)
  - Two stage: 25 mbar(a)
  - Three stage: 7 mbar(a)
  - Four stage: 1 mbar(a)
  - Five stage: 0.1 mbar(a)
- Capacity range
  - 25 mm to 2,500 mm inlet pipes
  - Up to 1,500,000 m³/h inlet gas flows
- Multiple elements for increased capacity
  - Standard sizes up to 1,000 mm inlet pipe

Edwards dry vacuum systems, liquid ring pump systems and EH boosters are ATEX certified up to Zone 0 while our other mechanical boosters are certified up to Zone 1.

7.7 APPLICATION ENGINEERING, DESIGN AND SYSTEMISATION

Edwards' application expertise and in-depth knowledge of the chemical process industry, based on thousands of installations, enable a comprehensive package of design and systemisation solutions.

- Optimisation of vacuum system design,
- Focus on safety, reliability, durability, efficiency and environmental considerations,
- Equipment selection, specification and integration into the process plant,
- Compliance with user requirements and specifications,
- Vacuum system integration with user control systems,
- Supervision of commissioning, installation and operator training.

Our highly experienced team of Application and Proposal Engineers, located in regions close to our customers, will assist with the selection, sizing and design of your vacuum pumping system to meet your vacuum needs. Meanwhile, our Engineers in the product companies will handle the order execution of your vacuum system from the receipt of order to the design, assembly and testing. Further, our team of Commissioning Engineers can support the installation on site.

7.8 HYBRID SYSTEMS

END-TO-END PROJECTS FOR PROCESS IMPROVEMENT

We have developed and delivered state-of-the-art technologies in various combinations to provide the most reliable and customised system solutions for our process customers. Our applications knowledge and vacuum expertise make it possible for us to provide the best combination of dry pumps, liquid ring pumps, mechanical boosters and steam ejectors with the lowest cost of ownership and highest reliability.

VARIETY OF DRY PUMP AND LIQUID RING PUMP COMBINATIONS TO PROVIDE THE MOST RELIABLE SYSTEM FOR YOUR PROCESS
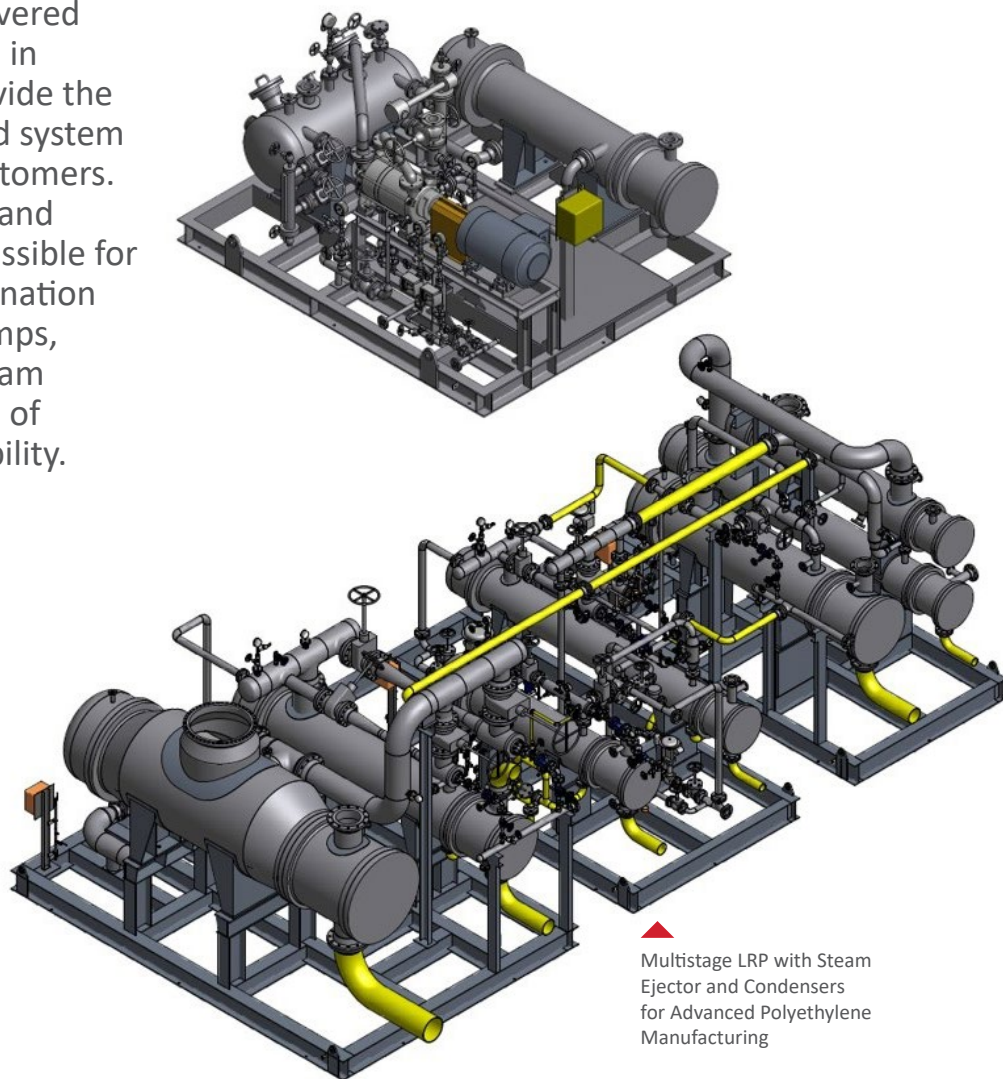
▲ Multistage LRP with Steam Ejector and Condensers for Advanced Polyethylene Manufacturing

▲ CDX100 Dry screw pump with EH2600 Booster package for chemical application
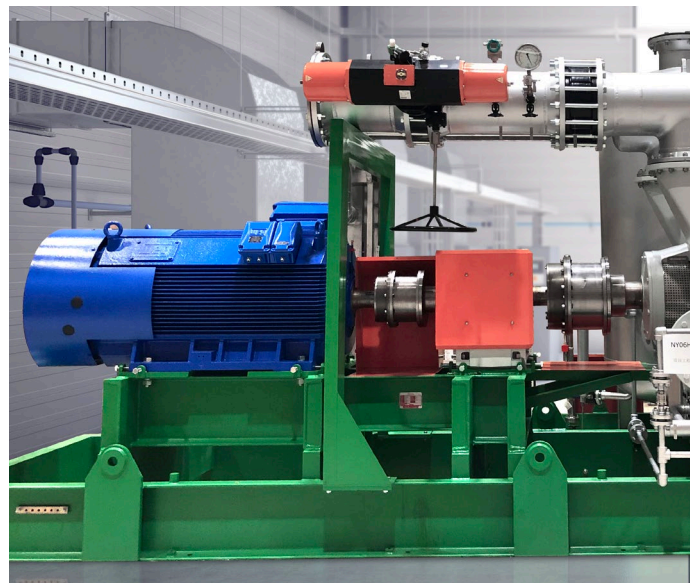
▲ Single-stage Liquid Ring Package
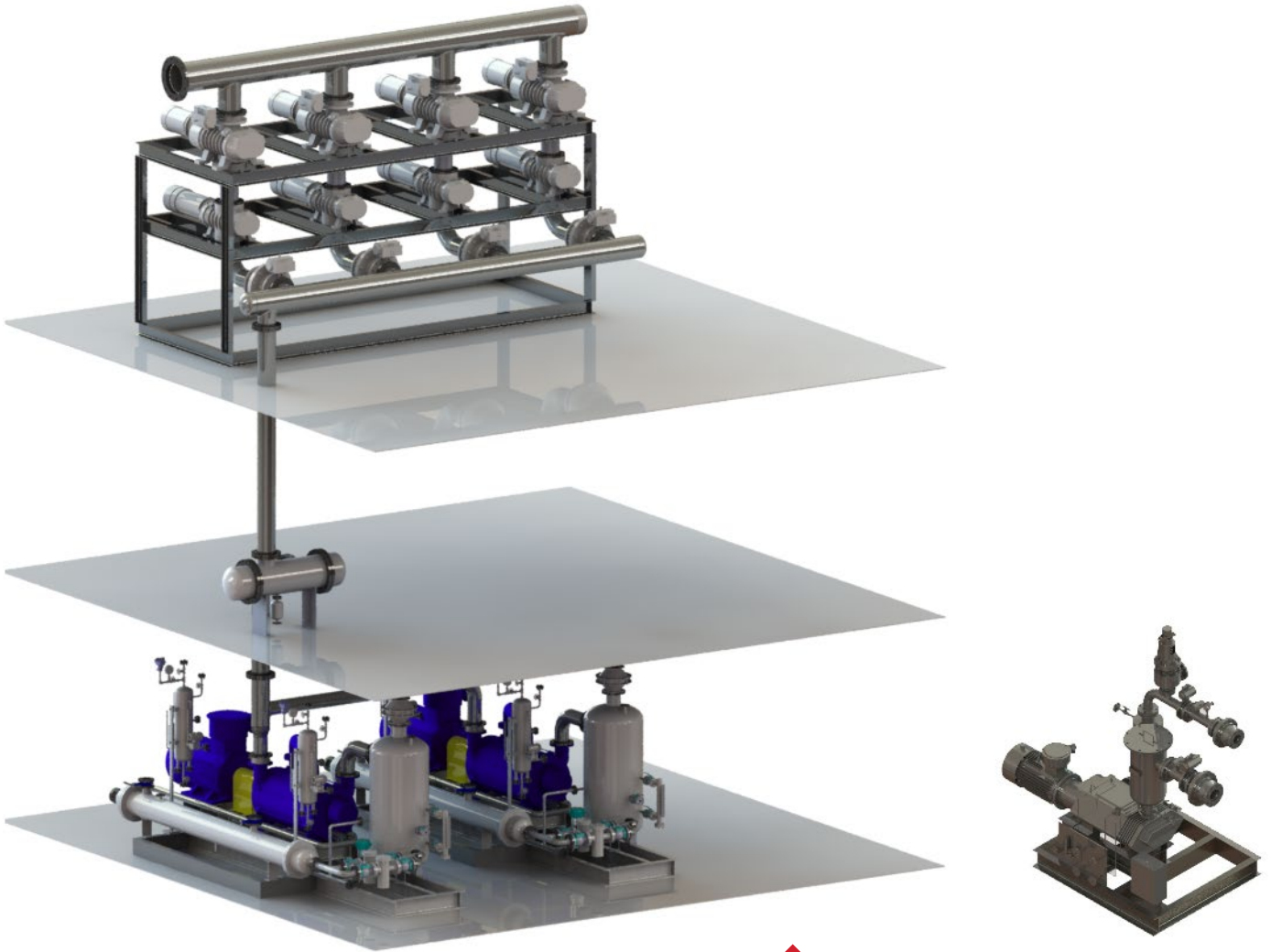
▲ Hybrid system with Stokes Booster,
CDX Dry screw pump and
Liquid Ring Pump combination for
DPC Polycarbonate application
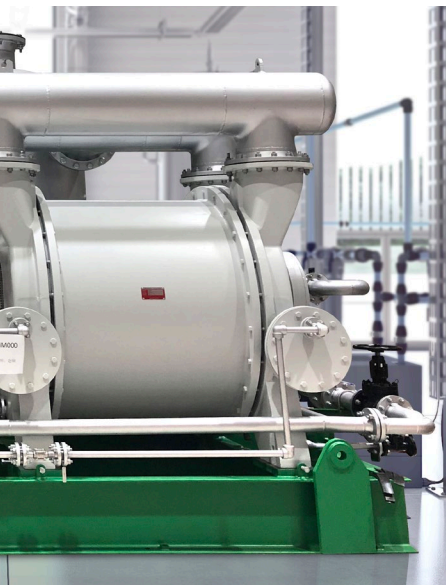
▲ Double Stage LRP for Condenser Air Extraction

8. SERVICE SOLUTIONS

Edwards understands the importance of local support. We have a number of major service facilities located around the world, each location supported by an extensive team of engineers and technicians to provide local, rapid response and great value service.

- Comprehensive service
- Extended warranties
- Repair
- Managed maintenance
- Certified products

All our service operations are conducted at the highest international standards in accordance with ISO 9001 (quality), ISO 14001 (environmental) and OHSAS 18001 (workplace safety).

9. UNIT CONVERSIONS

Pressure Units

	mbar	bar	Torr	Pa (Nm^{-2})	atm	lbf inch^{-2}
1 mbar =	1	1×10^{-3}	0.75	10^2	9.87×10^{-4}	1.45×10^{-2}
1 bar =	10^3	1	7.5×10^2	1×10^5	0.987	14.5
1 Torr =	1.33	1.33×10^{-3}	1	1.33×10^2	1.32×10^{-3}	1.93×10^{-2}
1 Pa (Nm^{-2}) =	0.01	1×10^{-5}	7.5×10^{-3}	1	9.87×10^{-6}	1.45×10^{-4}
1 atm =	1.01×10^3	1.01	7.6×10^2	1.01×10^5	1	14.7
1 lbf inch^{-2} =	68.9	6.89×10^{-2}	51.71	6.89×10^3	6.80×10^{-2}	1
1 kgf cm^{-2} =	9.81×10^2	0.98	7.36×10^2	9.81×10^4	0.97	14.2
1 inch Hg =	33.9	3.39×10^{-2}	25.4	3.39×10^3	3.34×10^{-2}	0.49
1 mm Hg =	1.33	1.33×10^{-3}	1	1.33×10^2	1.32×10^{-3}	1.93×10^{-2}
1 inch H$_2$O =	2.49	2.49×10^{-3}	1.87	2.49×10^2	2.46×10^{-3}	3.61×10^{-2}
1 mm H$_2$O =	9.81×10^{-2}	9.81×10^{-5}	7.36×10^{-2}	9.81	9.68×10^{-5}	1.42×10^{-3}

	kgf cm^{-2}	inch Hg	mm Hg	inch H2O	mm H2O
1 mbar =	1.02×10^{-3}	2.95×10^{-2}	0.75	0.40	10.2
1 bar =	1.02	29.53	7.5×10^2	4.01×10^2	1.02×10^4
1 Torr =	1.36×10^{-3}	3.94×10^{-2}	1	0.54	13.6
1 Pa (Nm^{-2}) =	1.02×10^{-5}	2.95×10^{-4}	7.5×10^{-3}	4.01×10^{-3}	0.10^2
1 atm =	1.03	29.92	7.6×10^2	4.07×10^2	1.03×10^4
1 lbf inch^{-2} =	7.03×10^{-2}	2.04	51.71	27.68	7.03×10^2
1 kgf cm^{-2} =	1	28.96	7.36×10^{-2}	3.94×10^2	1×10^4
1 inch Hg =	3.45×10^{-2}	1	25.4	13.6	3.45×10^2
1 mm Hg =	1.36×10^{-3}	3.94×10^{-2}	1	0.54	13.60
1 inch H$_2$O =	2.54×10^{-3}	7.36×10^{-2}	1.87	1	25.4
1 mm H$_2$O =	10^{-4}	2.90×10^{-3}	7.36×10^{-2}	3.94×10^{-2}	1

Note also: 1 dyn cm^{-2} (barye) = 0.1 Pa (Nm^{-2}) = 10^{-3} mbar

Leak Rate Units

	mbar ls^{-1}	Torr ls^{-1}	atm cm^3 s^{-1}	lusec	atm ft^{3}min^{-1}
1 mbar ls^{-1} =	1	0.75	0.99	7.5×10^2	2.09×10^{-3}
1 Torr ls^{-1} =	1.33	1	1.32×10^3	103	2.79×10^{-3}
1 atm cm^3 s^{-1} =	1.01	0.76	1	7.6×10^2	2.12×10^{-3}
1 lusec =	1.33×10^{-3}	1×10^{-3}	1.32×10^{-3}	1	2.79×10^{-6}
1 atm ft^{3}min^{-1} =	4.78×10^2	3.59×10^2	4.72×10^2	3.59×10^5	1

Pumping Speed Units

	ls^{-1}	l min^{-1}	ft^{3}min^{-1}	m^{3}h^{-1}
1 ls^{-1} =	1	60	2.119	3.60
1 l min^{-1} =	0.017	1	0.035	0.06
1 ft^{3}min^{-1} =	0.472	28.317	1	1.699
1 m^{3}h^{-1} =	0.278	16.667	0.589	1

10. REFERENCES

Jousten, Karl, [ed.]. *Wutz Handbuch Vakuumtechnik.* 11. edition, Berlin : Springer Vieweg, 2012.

BS3636. *Methods for proving the gas tightness of vacuum or pressurized plant.* 1963.

Mullin, J. W. *Crystallization.* 4th edition. Oxford : Butterworth-Heinemann, 2001.

A simple guide to vacuum measurement. Vacuum Science World.

Vacuum Pump Technologies. Vacuum Science World.

Essential guide to vacuum leak detection. Vacuum Science World.

Tighten up on vacuum leaks. Kursay, Roland. 1993, The Chemical Engineer.

Freeze drying. Garz, Dr. Juliane.: EDWARDS, 2019, Application Note.

ATEX. An Introduction.: EDWARDS, Application Note.

Processes and vacuum equipment in the chemical industry.: EDWARDS, 1997, Application Update.

Chemical Properties and Pump & Condenser Sizing. issue A: EDWARDS Internal, Technical Note.

Concentration, evaporation and crystallization. issue A: EDWARDS internal, 2005, Application Overview.

Filter drier systems. issue A: EDWARDS internal, 2004, Application overview.

The principle and application of gas balast.: EDWARDS, 2018, Application Note.

Vacuum Pump and Vacuum System Safety Manual. issue E: EDWARDS, 2019, Manual.

Vacuum Solutions for the Chemical Process Industries.: EDWARDS, 2020.

Vacuum Solutions for the Chemical Process Industries.: EDWARDS, 2016.

Solvent Recovery. issue A: EDWARDS internal, 2006, Application Note.

Chemical reactions. issue A: EDWARDS internal, 2005, Application Note.

Choosing Process Vacuum Pumps. Collins, Don.: AIChE, August 2012, Chemical Engineering Process, pp. 65-72.

Rawlinson, Phil. Module 2A- Technology the basics. *Liquid ring pump training programm*.: EDWARDS internal, 2020.

The technology of dry vacuum pumps.: Edwards.

edwardsvacuum.com

EDWARDS

Part of the Atlas Copco Group

GLOBAL CONTACTS

EMEA

UK	+44 1444 253 000
	0345 9 212 223
Austria & Switzerland	+49 (8)9 991 918 56
BeNeLux	+44 1293 60 3350
France	+33 (0)1 41 21 12 56
Germany	0800 000 1456
Italy & Slovenia	+39 02 48 44 71
Israel	+972 8 374 1111
Russia	+7 495 933 55 50 - Ext. 1800
Spain	+34 (9)1 65 32 185
Rest of Europe, Middle East & Africa	
	+420 (0) 533 441 045

ASIA PACIFIC

China	+86 400 111 9618
India	+91 20 4075 2222
Japan	+81 47 458 8822
Korea	+82 31 716 7070
Singapore	+65 6546 8408
Taiwan	+886 3758 1000

AMERICAS

USA	+1 800 848 9800
Brazil	+55 11 3952 5000