The vacuum oven in the laboratory

Functions, heating concepts and applications

Contents

1. Summary

Vacuum technology is used practically everywhere in industry and research. Vacuum grippers transport products from A to B, vacuum packaging protects perishable foods, LEDs are manufactured under vacuum and many everyday objects are coated under vacuum to be more robust and resilient. Vacuum drying covers a large area - whether as a large-scale system in the production of food and medicines or on a laboratory scale for drying, storing and degassing a wide variety of loads. In this whitepaper, we present the versatile laboratory vacuum oven in more detail.

To begin with, we unravel the mystery of what a vacuum is, where the term originated and since when the sciences deal with the theoretical and practical basics of vacuum technology. Then the advantages of vacuum drying are listed, and the components of a vacuum oven are described. In the last part, we delve into the most important applications of vacuum drying in the laboratory.

2. Vacuum in physics and practice

2.1. Origins of vacuum technology

> **"For by convention colour exists, by convention bitter, by convention sweet, but in reality atoms and the void."** Demokrit, after Galenus of Pergamum

Is there a vacuum and, if so, how can one create it? The origins of vacuum technology go back to antiquity. Democritus of Abdera (460 – 370 BC) and his teacher Leucippus are considered the fathers of the atomic theory. Democritus assumed that the world was made up of many small and indivisible particles, which he called atoms (atomos, Greek: indivisible). Between the atoms, Democritus suspected an empty space (a kind of micro-vacuum) through which move the atoms according to the general laws of mechanics. Variations in shape, orientation and arrangement of the atoms led to variations of macroscopic objects[1]. The idea of an empty space, a vacuum, was postulated for the first time. However, the two natural philosophers remained alone for many centuries. The influence was too great by Aristotle, who held the view that, in nature, there was a fear of emptiness (horror vacui) and therefore they would immediately fill any vacuum with surrounding matter.

Only with the advent of experimental physics in the 17th century did the vacuum come back into the focus of scholars. Galileo Galilei determined the specific gravity of air, thus proving that air is matter. His assistant, Evangelista Torricelli, succeeded in creating a vacuum with the help of a column of mercury and at the same time inventing the principle of the barometer. He filled a 1 m long tube with mercury and immersed it with the sealed opening down in a vessel with mercury. After removing the cap, the mercury tube sank, and a vacuum had formed at the top of the tube.

The explanation: The pressure of the mercury column counteracts the atmospheric air pressure from below – until equilibrium has been reached at a column height of around 760 mm. Blaise Pascal later demonstrated that the atmospheric pressure decreases with increasing altitude by repeating Toricelli's experiment at different points on a mountain.

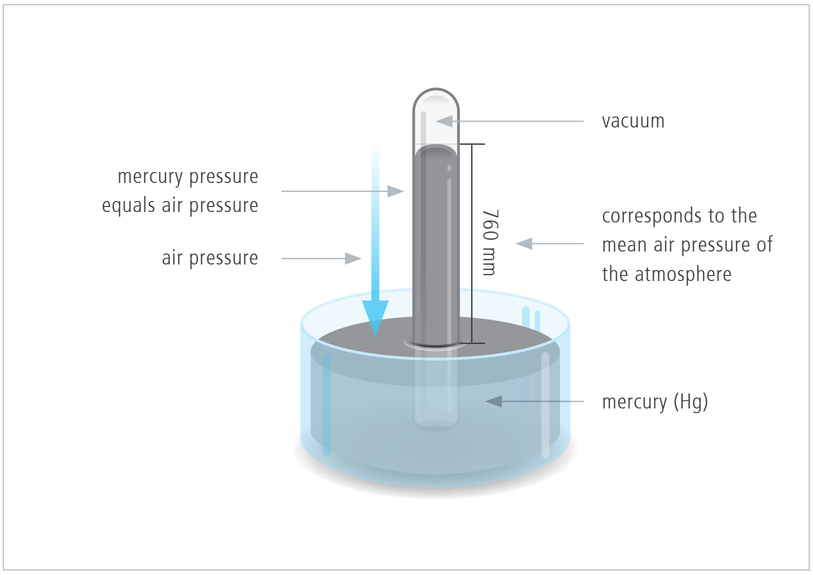

Figure 1: Torricelli's barometer
Figure 2: Original Magdeburg hemispheres including air pump (Deutsches Museum, Munich) from LepoRello/Wikimedia Commons. License: CC BY SA 3.0

Finally, the Mayor of Magdeburg, Otto von Guericke, demonstrated the first vacuum pump with his famous Magdeburg hemisphere experiment. He pressed two copper hemispheres together and pumped the air out through a valve with the piston air pump he had invented. Two teams of horses could not separate the hemispheres, which were only held together by air pressure, because there was no counteracting pressure on the inside.

2.2. What is vacuum?

The discussion as to whether there can actually be a completely empty space continues to this day. And so, one differentiates between the idea of theoretical physics of the absolute vacuum as a space without matter and the definition of practical physics. According to the latter, a vacuum is a space in which the pressure is less than the surrounding atmospheric pressure.

The definition in DIN 28400 Part 1 is: "Vacuum is the state of a gas when the pressure of the gas and thus the particle number density in a container is lower than outside or when the pressure of the gas is lower than 300 mbar, i.e., lower than the lowest atmospheric pressure occurring on the earth's surface."[2]

2.3. Atmospheric pressure

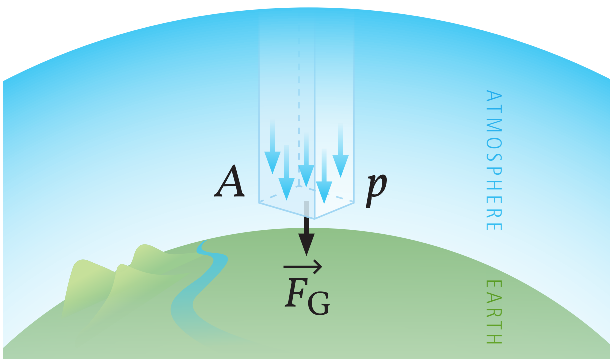

Figure 3: Atmospheric pressure illustration

Pressure is defined as force per area. A pascal is the pressure that a force of one Newton exerts on an area of one square meter.

$$1\mathrm{Pa} = 1\frac{\mathrm{N}}{\mathrm{m}^2} = 1\frac{\mathrm{kg}}{\mathrm{m}\cdot\mathrm{s}^2}$$

Above the earth is a gaseous atmosphere that is held in place by gravity. If we stand on a surface A on the ground, the weight $\vec{F_G}$ (physical unit Newton) of the air column above acts on our body.

The total mass of the atmosphere is about 5.13×10^{15} t, the surface of the earth is about 510 million km^2. Approximately, there is around 10,000 kg of air above every square meter of earth's surface. Since the altitude is just one of several influencing factors, along with the air temperature and the composition of the air, there is no exact mathematical formula for calculating the atmospheric pressure, only an approximation.

A simplified rule of thumb: decrease by 1% every 80 m or by 10% every 840 m.

The pressure loss with increasing height also affects applications in vacuum technology because the maximum achievable pressure difference decreases.

3. Advantages of vacuum drying

3.1. Heat-sensitive goods can be dried faster under vacuum

SUMMARY

The ability to lower the boiling point of liquids in a controlled manner is a key user advantage of a vacuum oven. On one hand, the drying (material transfer) is accelerated, on the other hand, heat-sensitive materials are protected. Heat changes the structure, color or aroma of most organic foods, proteins denature or the nutrient and vitamin content decreases. The effectiveness of pharmaceutical products can be impaired, and electronic components react with a loss of function and some plastics can outgas or become brittle due to heat. The low oxygen content in the vacuum oven also minimizes oxidation and corrosion processes. In order to remove oxygen almost completely (to avoid oxidation), an inert gas (e.g., nitrogen) can be introduced into the interior.

At sea level, water boils at around 100 °C and begins to evaporate. Air pressure decreases exponentially with altitude. If the air pressure drops below normal pressure, the boiling temperature also drops and vice versa. As a result, water on Mount Everest only boils at around 70 °C, and it takes much longer to boil than in a pressure cooker, which reaches temperatures approaching 120 °C at 1.8 times normal atmospheric pressure.

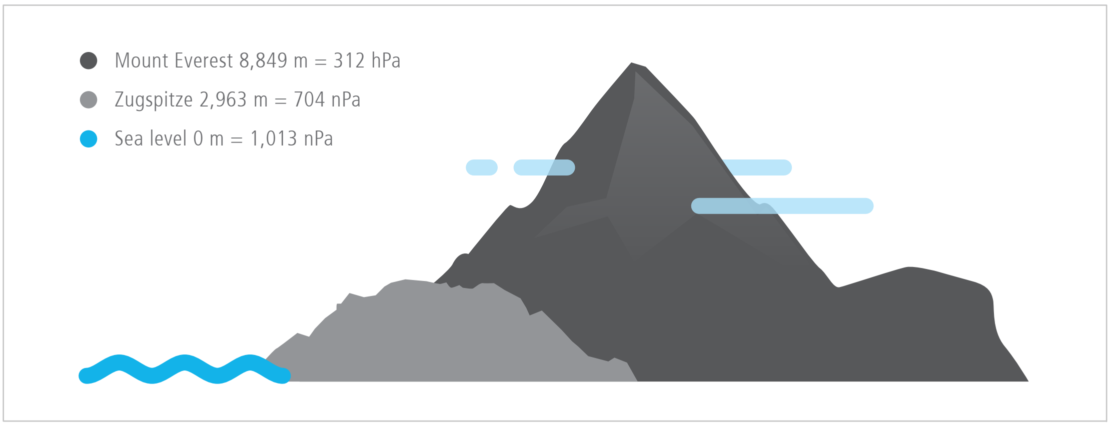

Figure 4: The boiling temperature of water is internationally set at 100 °C for a mean air pressure at sea level of 1013.25 hPa (normal pressure). At 312 hPa, the air pressure on Mount Everest is just under a third of normal pressure, which is why water boils at around 70 °C

3.2. Extremely low residual moisture is possible under vacuum

SUMMARY

In the vast majority of cases, we are dealing with capillary-porous goods in industrial drying. They attract moisture from the air and bind water from the environment in their pores, which are connected to each other and to their environment, until the water is saturated. The material to be dried, so to speak, has an outer & an inner surface that needs to be dried. Since the water molecules in the vacuum have to overcome a lower gas pressure in order to transition into the gas phase and the gas pressure acts uniformly everywhere, goods can be dried more reliably and quickly in the vacuum to zero residual moisture. The latter is also a significant advantage for technical components with complex geometries or undercuts.

"Drying is the separation of liquid from a solid-liquid system by evaporation or evaporation of the liquid. (...) The resulting vapors - called vapors - are either discharged through the dry air or drawn off by pressure or partial pressure gradients. Evaporation is carried out at normal pressure or negative pressure."[3]

In thermal drying processes, this occurs through what is known as the phase change from the liquid phase to the gas phase (see Figure 4). The respective state of aggregation depends on pressure and temperature.

Water is solid (ice) at normal pressure (1013 hectopascals, hPa) and at temperatures below 0 °C, liquid between 0 °C and 100 °C and gaseous (steam) at the boiling point, i.e., at temperatures above 100 °C. "The special case of boiling, evaporation [note below the boiling point] occurs when particles with a high kinetic energy (e.g., due to the influence of heat) can

[1] An inert gas (inert gas, protective gas) is a gas that does not react in the respective reaction system. Inert gas is primarily used to displace or dilute other reactive gases, especially oxygen. Source: www.chemgapedia.de

overcome the attractive forces of particles with lower kinetic energy in a liquid. If the particles remaining in the cooled liquid continue to draw energy from the environment, they evaporate the liquid is complete at the end."[4]

In the vicinity of liquefaction, gases are referred to as vapour. The amount of energy required for the phase transition – reaching the boiling point – results from the substance-dependent vaporization enthalpy. This is particularly high in water, since the water molecules are light on the one hand and attract one another particularly strongly on the other.

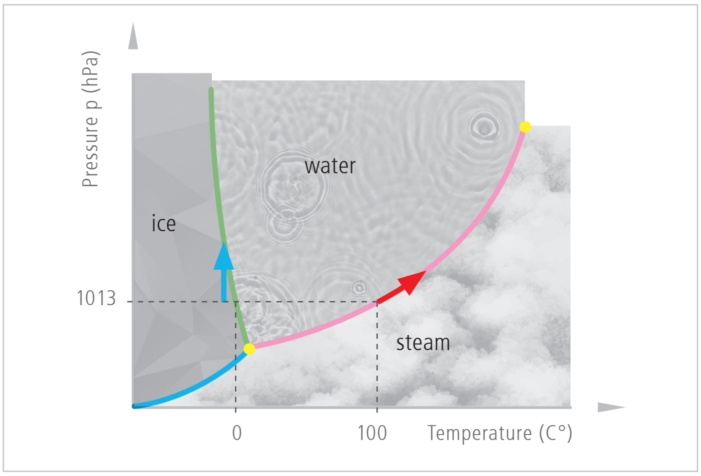

Figure 5: Phase diagram of water

For the description of the drying process, a distinction is made between two main forms of how the moisture is mechanically bound to the solid (in addition, there is also sorptively bound moisture).[4]

1. Freely attached surface moisture

A saturated water vapor-air mixture forms directly above the surface moisture. In this case, the vapor partial pressure corresponds to the saturation vapor pressure of the surface water. The fact that water can also evaporate in a vacuum shows that the saturation concentration is essentially dependent on the kinetic energy of the water particles described above, i.e., the temperature of the surface water. The driving force for the evaporation of the water vapor is the pressure gradient, because if one of the parameters temperatures (via the contact surface of the heater) or partial pressure (via pumping the moist air out of the interior of the vacuum chamber) changes, the entire system strives to restore equilibrium. If the surface (as well as pores and capillaries with large diameters) has dried, the remaining liquid is only in the gut interior.

2. In the capillaries or pores of the dry material

This moisture is transported to the surface of the goods during drying by capillarity or diffusion. Additional energy is required to overcome the capillary forces in the fine capillaries. In this second phase of the drying process, in particular, vacuum technology has decisive advantages: since the lower pressure acts simultaneously everywhere on the drying goods, the residual moisture evaporates more quickly, even in places where classic convection drying has trouble.

[2] The enthalpy of vaporization is the energy required to vaporize a given amount of liquid at a given temperature. Its amount is equal to the amount of the corresponding enthalpy of condensation with the opposite sign. | [3] Dalton's law of partial pressures (1801) states that the total pressure p of a mixture of ideal gases is equal to the sum of the partial pressures of the individual gas components pi. Source: www.chemgapedia.de | [4] The pressure at which the liquid and gaseous phases coexist is called the vapor pressure (symbol pD). It increases exponentially with temperature. Source: www.chemgapedia.de | [5] Capillarity [from *capillary-], rise in wetting or fall in non-wetting liquids in capillaries (capillaries) or at the edge of the vessel. The interaction of cohesion and adhesion forces causes the surface of the liquid (meniscus) to curve either upwards or downwards, depending on whether the molecules are attracted more towards each other or towards the vessel wall (surface tension). Source: www.paket.de

3.3. Heat-sensitive goods can be dried in a vacuum in an energy-efficient manner

The vacuum pump is the largest energy consumer in a vacuum oven, followed by the heater. Intelligent control technology is therefore crucial for energy-saving operation. Speed-controlled pressure control, in which the pump output always follows the actual vacuum requirement, has proven to be advantageous in practice. The direct heating of the load using heated plates or trays also reduces the energy requirement, as the drying process is accelerated (see also the chapter on direct heating with multi-level sensing & heating technology, page 10).

4. Components of a vacuum oven

SUMMARY

The gas pressure in the vacuum cabinet is reduced by gas particles being removed from the interior ①. The gas is generally evacuated using directly connected membrane vacuum pumps ② or a central vacuum system. In order to prevent saturation of the interior air in the case of very damp goods or to transport the moisture away more quickly, the interior can be flushed with fresh air. The working space therefore has connections for ③ the gas outlet and the supply of fresh air or inert gas. The heat is transferred by conduction. The items to be dried stand on heated surfaces ④. The heating takes place either indirectly with an air jacket heater or directly with a plate heater. Pressure and temperature can be set in a controlled manner via digital controls for vacuum pump ② and heater ④.

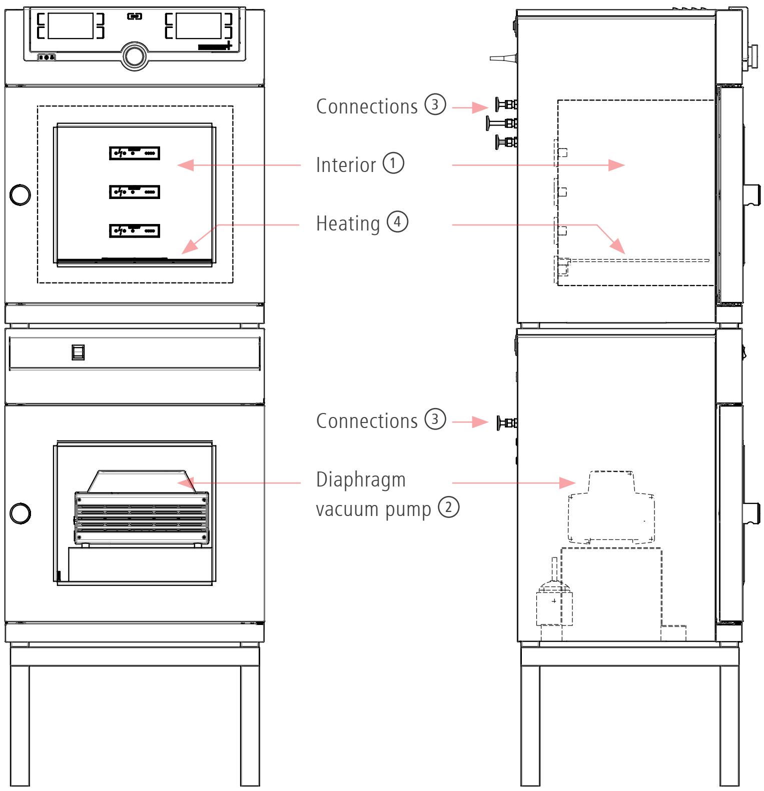

Figure 6: Components of the Memmert vacuum oven VO

4.1. Interior

Vacuum ovens for use in the laboratory usually have an interior volume of between 20 and 150 liters and a temperature range starting between +5 °C and +15 °C above room temperature to +250 °C; some devices can also be operated up to 400 °C. The smaller the working space, the faster the ideal printing conditions can be adjusted. Interiors are mostly made of rust-free, acid-resistant stainless steel 1.4301 (according to the ASTM 304 standard) or the more resistant 1.4404.

The low boiling temperatures and pressures also affect the safety of people, dry goods and equipment. Safety glass doors are therefore part of the equipment of every high-quality vacuum oven.

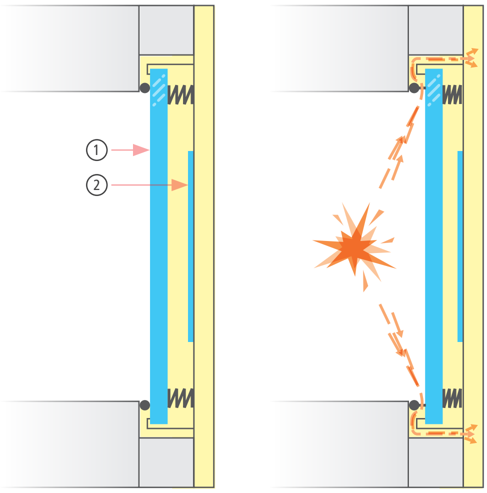

Figure 7: Safety glass doors using the example of a Memmert vacuum oven VO: 15 mm thick safety glass ① spring-loaded on the inside, door outside with splinter protection pane ② - any sudden overpressure escapes via the spring-back safety glass pane

4.2. Comparison of jacket heating vs. shelf heating

In the almost airless space, there are not enough gas particles to be able to conduct heat. Therefore, the material to be dried is heated through direct contact with a heated surface. Why is good heat transfer so important? When liquid evaporates, heat is extracted from the charge. This is colloquially called "evaporative cooling". Very moist loads would cool down so much due to the heat withdrawal that they would even freeze. The low temperatures in the vacuum also slow down the drying process, since the boiling point of the liquid is not reached. Therefore, the heat losses must be compensated for quickly by good heat transfer from the heated surface to the load. Important here: the largest possible surface of the goods rests on the heater without an "air gap".

Two main heating systems are used for contact drying in a vacuum oven: jacket heating, in which the heat is conducted from the side walls of the chamber to carrier plates, or shelf heating, in which the carrier plates are heated directly. Jacket heating has the disadvantage that adjusting the temperature takes significantly longer than with direct heating of the heat exchanger. In the case of shelf heating, the fixed installation of the support plates and the lack of the possibility of separate heating control for each shelf can be a disadvantage in some models.

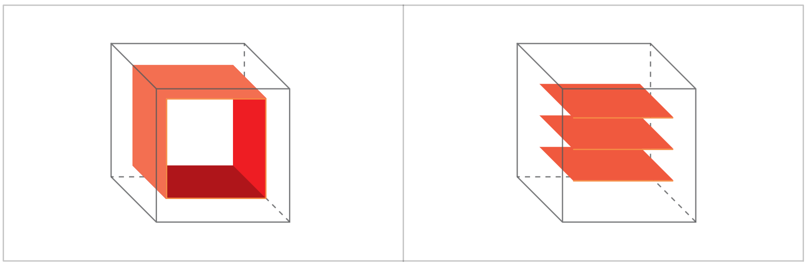

Figure 8: Jacket heating (left) and shelf heating (right)

The Memmert solution

Direct heating with Multi-Level-Sensing & Heating technology ensures the shortest possible heating and process times: thanks to a plug-in contact, each thermoshelf can be positioned individually, has a separate temperature controller and can be calibrated individually. The control circuits react precisely to different product temperatures (keyword "evaporative cooling") or product moisture and maintain the target temperature evenly during drying.

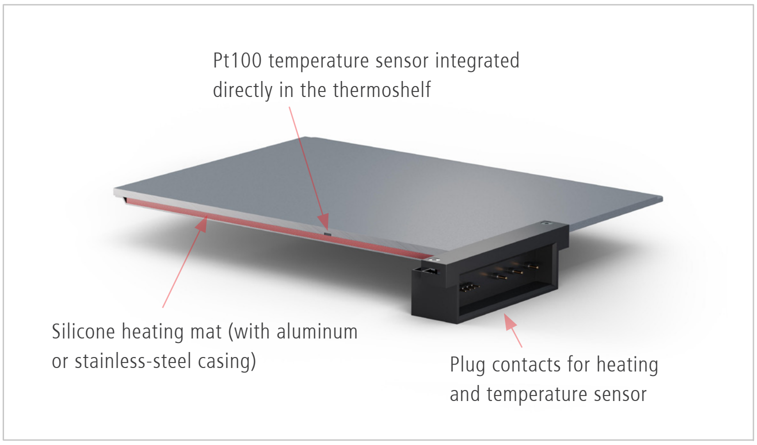

Figure 9: Memmert Multi-Level-Sensing & Heating Technology

Digitally controlled vacuum cycles significantly reduce drying time. With this function, the working area is ventilated at short intervals. Ramps with different temperature and vacuum setpoints can be easily programmed using the AtmoCONTROL software.

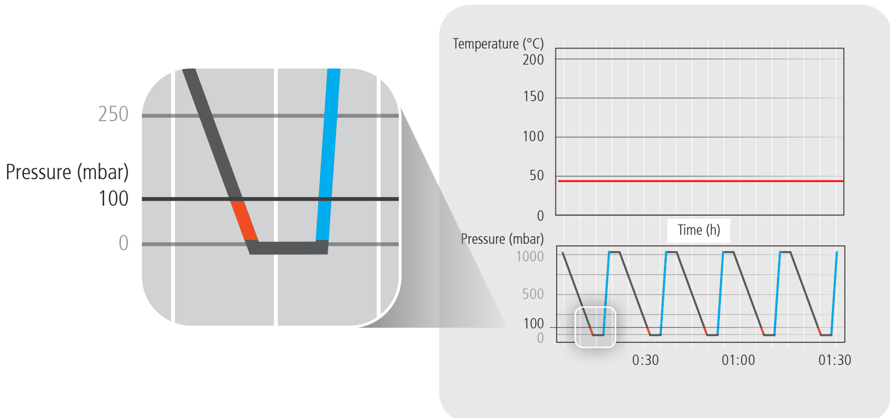

Figure 10: Turbo drying thanks to digitally controlled vacuum cycles

4.3. Functional principle of the diaphragm pump

Depending on the working pressure, a distinction is made in vacuum technology between rough vacuum, fine vacuum, high vacuum and ultra-high vacuum (maximum vacuum). As a rule, the pressure ranges of a vacuum drying oven for laboratory use range from normal pressure to rough vacuum. In a Memmert vacuum oven, for example, vacuums can be regulated between 5 mbar and 1100 mbar - the actual maximum corresponds to the ambient pressure. Devices in this pressure range are usually operated with electric diaphragm pumps unless the ovens are connected to a central pressure supply.

Pressure area	mbar	Molecules per m^3	Examples from technology and laboratory
Normal pressure	1013	2,7 to 10^{19}	–
Vacuum	1013 to 300	2,7-10^{19} to 10^{19}	Vacuum cleaner > 500 mbar
Rough vacuum	300 to 1	10^{19} to 10^{16}	Factory vacuum > 130 mbar, Membrane pump > 2 mbar
Fine vacuum	1 to 10^{-3}	10^{16} to 10^{13}	Rotary vane pump > 1 to 10^{-3} mbar
High vacuum (HV)	10^{-3} to 10^{-7}	10^{13} to 10^{9}	Oil diffusion pump > 1 to 10^{-5} mbar
Ultra-high vacuum (UHV)	10^{-7} to 10^{-12}	10^{9} to 10^{4}	Turbo molecular pump, cryopump

Figure 11: Reaching of different pressure ranges

Source: [3] aprentas (eds), page 179

Diaphragm pumps work according to the displacement principle, i.e., the working space changes in volume (see figure 12). The vacuum is present on the inlet side of the working chamber, and the sucked-in gas is discharged on the outlet side. To do this, a membrane above the working area is made to vibrate. The volume increases during the suction stroke and decreases during the delivery stroke (pumping stroke). The chambers are mechanically sealed, and the gas is transported via inlet and outlet valves. In order to increase the suction power, several chambers are usually connected in series in a diaphragm pump.

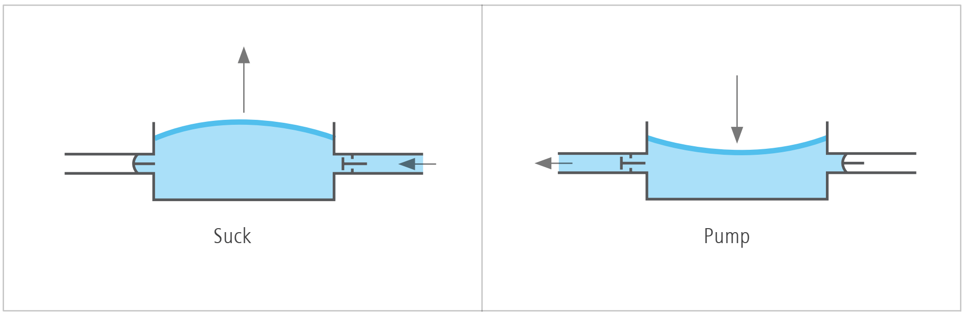

Figure 12: Displacement principle in diaphragm pumps

In addition to quiet operation and easy maintenance, important quality features of diaphragm pumps for use in vacuum chambers are chemical resistance and energy efficiency. A diaphragm pump can be operated in an energy-efficient manner with speed control. In the case of a displacement pump, a change in the speed means that the strokes are carried out more or less often accordingly.

The Memmert solution

Thanks to the infinitely variable speed-controlled vacuum pump, the VO series saves around 70% energy in ramp operation compared to an uncontrolled pump. The efficiency-optimized pump control ensures that the pump goes into standby mode or switches off completely when there is no need to pump. The simultaneous optimization of the rinsing processes increases the service life of the membranes.

5. Exemplary applications in the vacuum oven

5.1. Drying in vacuum

RESIDUE-FREE DRYING OF POWDER AND GRANULES
Industries: metallurgy, pharmacy, food industry, plastics industry

Advantages:
Faster removal of moisture

Challenge: When large amounts of powder and granules with a high moisture content have to be dried in the vacuum oven, the suction power of the vacuum pump reaches its limits at times. This is because water in the vapor phase has approximately 1673 times the volume, depending on temperature and pressure. In addition, the vacuum pump can transport the gas particles away better and faster at higher pressures than at lower pressures. A simple picture helps to understand: The vacuum pump is like a soup ladle. It can draw from the full, as long as the pot is well filled. The lower the filling level, the less content is transported away with one scooping process. The result: the supply of heat and the removal of water vapor become unbalanced, the samples dry extremely slowly or even boil in their own juice.

Solution: Controlled intermediate ventilation of the interior transports the moisture away more quickly. The Memmert vacuum oven VO offers the programming of vacuum cycles. This allows two vacuum values to be set, between which the pressure in the interior is regulated in a controlled manner. Within a short period of time, a large amount of dry air or inert gas is let in, which absorbs the moisture and is sucked off by the vacuum pump in the next vacuum cycle.

DRYING AND STORAGE OF ELECTRONIC COMPONENTS,
MACHINE COMPONENTS AND PRECISION MECHANICAL COMPONENTS
Sectors: contract assembly, manufacture of electronic and electromechanical assemblies, jewelry and watchmaking industry, industry

Advantages:
Shortening of the drying time
Reduction of the drying temperature
No residual moisture
Prevention of oxidation

Challenge: The base material of most printed circuit boards, especially flexible and rigid polyimide material, is extremely hygroscopic. This means the circuit boards absorb moisture from the ambient air and bind it in the lower layers. During the subsequent soldering process, this moisture can suddenly evaporate and lead to delamination of the layers, blistering, microcracks due to oxidation or malfunctions of the circuit board. Therefore, the drying of printed circuit boards before assembly is recommended or prescribed in the relevant standards.

In addition, the high temperatures involved in lead-free soldering led to aging of heat-sensitive surfaces such as, for example, chemical tin. Last, but not least, contaminated circuit boards and assemblies must be cleaned and then gently dried.

Solution: In a vacuum, heat-sensitive electronic components and metal components susceptible to corrosion be dried and stored at lower temperatures without leaving any residue. The drying time is also usually significant shortened.

Note: The explanations for drying electronic components in a vacuum are only of a general nature. In the specific case coordination with the manufacturers of printed circuit boards, adhesives, etc. must always take place.

GENTLE DRYING OF BIOLOGICAL AND ORGANIC CHEMICAL SUBSTANCES
Sectors: food industry, biological and chemical research

Advantages:
Protection or preservation of the cell structure
Less loss of nutrients
Less big change in external characteristics

Challenge: Many organic substances are sensitive to air and heat.

Solution: The lower boiling temperatures protect the substances, the vacuum prevents (possibly in combination with the use of inert gas) oxidation processes.

LOW TEMPERATURE VACUUM DRYING
Sectors: food industry, pharmaceutical industry

Advantages:

Gentle drying at low temperatures above freezing

Challenge: As a rule, probiotic bacterial strains and starter cultures are freeze-dried in order to be stable until they are added, i.e. first deep-frozen and then dehydrated under vacuum. In practice, however, this method has two major disadvantages: it is energy-intensive, and, in addition, some strains of bacteria cannot survive temperatures below freezing.

Solution: This drying process allows labile fabrics to be dried at moderate temperatures but above freezing dried without damaging the cell structure too much. In cooperation with the Technical University of Munich, Memmert has developed a low-temperature vacuum drying oven with Peltier cooling, which opens up new opportunities for research in the food and pharmaceutical industries. For example, program-controlled, controlled transport and storage scenarios can be simulated in order to determine changes in active ingredients or volumes under different pressure and temperature conditions.

5.2. Determination of dry matter or moisture content

QUALITY CONTROL
Sectors: pharmacy, food industry, environmental technology

Advantages:

Faster determination of the moisture content of biomass as well as medicinal and food products containing moisture

Challenge: For many industrial and scientific applications, it is necessary to determine the moisture content or the dry matter of samples as part of quality assurance. Whether and when the so-called drying oven method has to be used is often regulated by standards. However, the classic drying processes take a long time.[6]

Solution: In many cases, vacuum drying can significantly shorten the process of determining dry matter. AOAC (Association of Official Agricultural Chemists) International officially recognizes vacuum drying as a method for many foods.

[6] The dry box method is used to measure the moisture content of a material/sample. It is a measuring method of gravimetry, more precisely thermogravimetry, in which the content of liquid and volatile substances is determined via the mass. A precisely weighed sample is exposed to a specific drying time and temperature. The loss on drying is then determined by re-weighing and the percentage moisture content is obtained. Source: https://www.memmert.com/products/heating-drying-ovens/

5.3. Degassing in vacuum

REMOVAL OF GASES AND VOLATILE SUBSTANCES
Sectors: electronics industry, plastics industry, food industry

Advantages:

Avoidance of air pockets and oxidation

Challenge: When casting thermosets, one usually wants to avoid pore formation due to air inclusions. In the electronics industry, the encapsulation of components with epoxy resins is a common method for insulation or to protect the components. If air bubbles become trapped during this process, this can lead to tension and lead to cracks and reduce the insulating effect. Another application is the removal of odorous and potentially harmful substances to prevent them from escaping uncontrolled into the environment during use.

Solution: Before curing resins and other thermosets, gases and other volatile substances such as solvents are removed in the vacuum oven at sub-atmospheric pressures. In the food industry, degassing (of oxygen) is used to avoid oxidation.

Sources:

[1] K. Jousten, "History of Vacuum Technology," in Handbuch Vacuum Technology, Wiesbaden, Springer Vieweg, 2018.

[2] DIN 28400-1 vacuum technology; terms and definitions; General designations, 1990.

[3] W. Hemming, Process Engineering, Würzburg: Vogel Buchverlag, 1986.

[4] "Temperature control units" in Basic Knowledge Laboratory Technology, Berlin, VGKL, 2012, pp. 321-337.

[5] G. E. Dieter Gehrmann, "Chapter 3 Introduction to the principles of drying technology," in Trocken, Aulendorf, ECV Editio Cantor, 2011, pp. 20-116.

[6] a. (eds), "Working with Vacuum," in Laboratory Practice Volume 1: Introduction, General Methods., Cham, Springer, 2017, pp. 177-186.

Figure 2: https://commons.wikimedia.org/wiki/File:Magedurger_halbkugeln_luftpumpe_Deutsches_Museum.jpg

Now in its third generation, Memmert has been developing and producing climate chambers, heating and drying ovens, incubators, medical devices as well as water baths at two locations in southern Germany (Schwabach and Buechenbach) for a very wide range of applications. Around 500 employees from about 30 nations are involved in the success of our company. In over 190 countries all over the world, hundreds of thousands of Memmert products have been permanently in use for decades. Therefore, Memmert is one of the most innovative and leading manufacturers of climate and temperature control devices worldwide.