

ROADMAP...

...FOR VACUUM IN THE LABORATORY

Technology and applications for chemistry and life science



Technology for Vacuum Systems

FOREWORD

Unbelievable but true, a technology with which you literally produce “nothing” belongs to a widespread key technology. Whether at home whilst vacuuming in the manufacture of various goods, in automation or in the laboratory, vacuum technology is used everywhere. On the following pages we will focus on the laboratory and provide you with an overview of the special requirements that vacuum technology fulfils and the technical solutions that are most appropriate.

“The right tool saves time” – this old saying of craftsmen applies not only to the building site, but also in the laboratory. Precisely because it is so intangible, the importance of the role of vacuum is frequently underestimated. But for many laboratory applications, it is invaluable. Clever vacuum technology allows you to achieve desired results faster, more safely and more conveniently. For this reason, for more than 50 years, we have dedicated ourselves to vacuum technology in the laboratory and are proud of the fact that we are able to offer the most comprehensive and advanced product range for this purpose. In this brochure, we will present you with important distinguishing characteristics that you should consider in order to find the right vacuum solution for your laboratory and application needs.

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WHY VACUUM?

Many people use vacuum every day in the laboratory. But what is the actual purpose behind it? Vacuum is used for many standard applications in the preparation and processing of samples. In most cases, the vacuum is not the focus, but it is absolutely essential. The most well-known applications for vacuum are vacuum filtration and drying.

Of course, you could filter without vacuum as well - like brewing coffee - by letting gravity do the work for you. However, whilst this may work for coffee, it does not always work in the laboratory like this because of the broad spectrum of solvents and solid substances used there. To speed up the process, low-pressure - i.e. vacuum - is created in a filter flask. The pressure difference in between the inside of the flask and the environment causes a perceived suction effect, causing the sample to flow through the filter more rapidly.

However, on a drying process the aggregate state of the sample changes from liquid to gaseous. Drying we could simply let happen in the air as we are used to dry our laundry, but since this would require too much time again, vacuum helps to accelerate this drying process. Under vacuum, less of compressive force acts on the molecules which enables the transition from fluid to gaseous condition more rapidly. In conclusion this physical phenomenon is the reason for that with decreasing of pressure level, less of heat energy is necessary to evaporate solvents. Therefore, the processing of heat-sensitive sample materials is only possible with the aid of vacuum.

Vacuum now then takes care for performing processes in laboratory more rapidly and more gently.



REQUIREMENTS FOR VACUUM TECHNOLOGY

The requirements for a vacuum are always dependent on the individual application and the use of very different solvents and substances. Characteristics such as boiling point, corrosion hazard and the quantity of the solvent to be evaporated play an important role in the selection and size of the appropriate vacuum equipment. For example, whether it is Methanol, Dimethyl Sulfoxide (DMSO) or a complex mixture that is to be evaporated at a certain temperature presents a critical difference since these substances all have different boiling points. Therefore, depending on the type of application, there are different requirements for the creation, measurement and regulation of the vacuum.

Ultimate vacuum and aspiration capacity

A vacuum pump is characterized by two essential characteristics: the lowest attainable pressure - also called ultimate vacuum - and the flow rate (pumping speed).

The ultimate vacuum is often given in millibar. The lower the value, the stronger, higher or better the vacuum. In the chemistry and life science laboratory, the required pressure range is usually down to about 10^{-3} mbar. Between one atmosphere (~ 1000 mbar) and 1 mbar, we speak in terms of a "rough vacuum", while the range between 1 and 10^{-3} mbar is commonly referred to as "fine vacuum". In the physics laboratory, we often encounter the application of pressures below 10^{-3} mbar as well. These pressure ranges are known as high vacuum and ultra-high vacuum (Fig. 1).

Depending on the respective pressure range, different pump technologies are used for vacuum creation. While the rough vacuum range can be covered most efficiently with diaphragm pumps, rotary vane pumps are frequently used in the creation of fine vacuum.

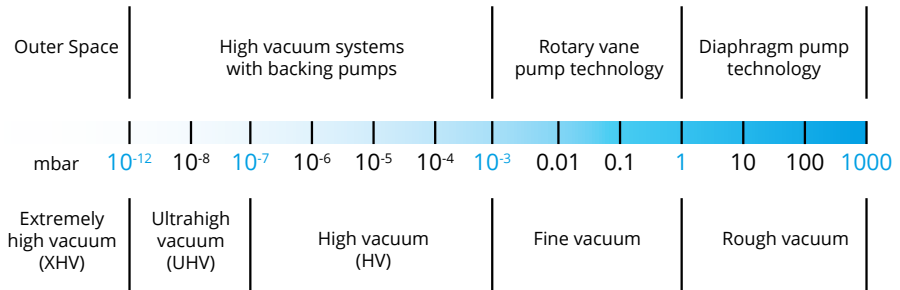


Fig. 1: Pressure ranges and technologies

Applications of fine vacuum

- ✦ freeze-drying
- ✦ residual drying in Schlenk lines
- ✦ molecular distillation
- ✦ evacuation of the sample chamber in analysis devices

Applications of rough vacuum

- ✦ filtration, suction
- ✦ solid phase extraction
- ✦ fluid aspiration
- ✦ solvent degassing
- ✦ vacuum concentration
- ✦ drying of protein gels
- ✦ drying of substances in a drying cabinet
- ✦ evaporation by means of rotary or parallel evaporators

The pumping speed is indicated in cubic meters per hour [m^3/h] or litres per minute [l/min] ($1 \text{ m}^3/\text{h} \triangleq 16.6 \text{ l}/\text{min}$). The greater the pumping speed, the faster the pump can evacuate a certain volume. When comparing the maximum pumping speed of two pumps, this is always measured under atmospheric pressure conditions, because as the pressure reduces, the pumping speed is also reduced since there are fewer and fewer molecules that can be “evacuated”. The extent of this loss of performance differs from pump to pump and very much depends on the pump technology used. If the pumping speed is too low at low (absolute) pressure, there is potential for significant negative consequences. First, low pumping speed at low pressure leads to extended evacuation times. Secondly, the pump may not be able to reach its rated ultimate vacuum if there are even small leaks in the system. Such a pump does not have enough power to compensate for the leak. Since leaks can never be completely avoided, it is the pumping speed at the desired process vacuum which is critical, and not the maximum value at atmospheric pressure ($\sim 1000 \text{ mbar}$) as specified in the data sheet. Therefore, you should always look at the pump curve to compare the pumping speed at actual process vacuum conditions. This represents the pumping speed as a function of the pressure. This curve will allow you to easily see how powerful the pump is in the range of the desired process vacuum.

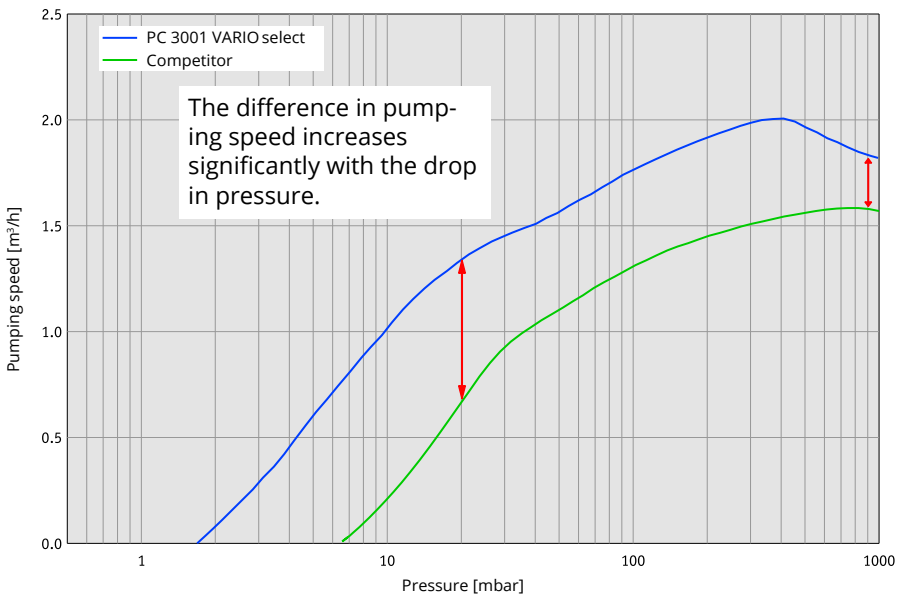


Fig. 2: Comparison of pump curves: PC 3001 VARIO select vs. competitor's product

Designed for demanding chemical processes

Using corrosion-resistant pumps is critical in chemistry labs. This is guaranteed first and foremost by the use of chemical-resistant plastics and special pump head technology.

Work in chemistry labs typically involves flammable solvents. To ensure safe operating conditions, most VACUUBRAND chemistry diaphragm pumps have a Category 3 ATEX rating on the inside parts that come into contact with the media. These pumps can be used without problem to support normal lab-scale work with nearly all common lab solvents

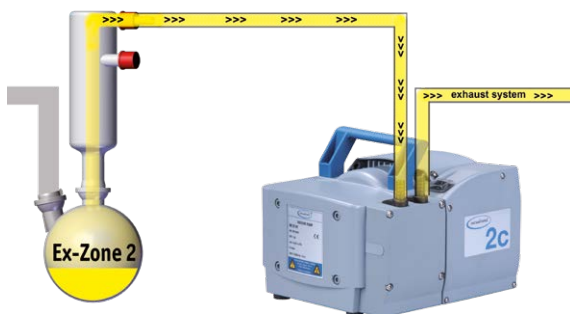


Fig. 3: Ex-Zone 2,
internal atmosphere

Precision and comfort

In order to carry out reaction sequences and processes in the laboratory in a controlled and reproducible manner, the pressure in the reaction vessel is measured and actively regulated depending on the application. A classic example is evaporation in a rotary evaporator. The vacuum generated must first correspond as accurately as possible to the boiling pressure of the solvent to be evaporated. If the pressure is too high, the process is unnecessarily long; if it is too low, the mixture to be evaporated can be caused to foam up and boil over, resulting in the sample being lost and then the evaporation process must be repeated.

The intelligent regulation technology of the VACUUBRAND VARIO® pumps makes the user's work simple here. This technology can determine and adjust the vacuum automatically in such a way that even in sensitive processes, foaming over does not occur and solvent mixtures are optimally and quickly evaporated. In this way, not only the desired results are achieved, but also it eliminates the need for unnecessary monitoring of the application and the process can be reproduced without problems at any time.



TECHNOLOGY

There are a variety of technical solutions for the requirements just described. At first glance, these differ based on the pump technology used as well as the number and connection of the pump heads. Here, the term pump head refers to the chamber in the pump through which media are pumped, and the adjacent mechanical components (Fig 4). However, the performance and robustness of a pump is also highly dependent on the construction and quality of the materials used. Additional distinguishing features arise from the accessories used. There are significant quality differences, particularly in the field of regulation technology. The following section provides an overview of the most important technical features and their functions.

Diaphragm or rotary vane pump?

As already mentioned, diaphragm pumps are usually used in the rough vacuum range and rotary vane pumps in the fine vacuum range. Basically, in a diaphragm pump, one or more diaphragms are moved up and down so that the pump chamber gets larger and smaller, thus producing a pumping effect. The diaphragm hermetically seals the pump chamber (in which the gases and vapours are aspirated and compacted) from the drive with the motor (airtight) (Fig. 4). The pump chamber is therefore completely dry (no operating materials / lubricants) and the extracted gases are not contaminated. Two mechanical valves ensure that extracted gas is aspirated from the correct tube and then ejected into the other. Thus, gas flow from the sample vessel through the pump in the direction of the exhaust is ensured.

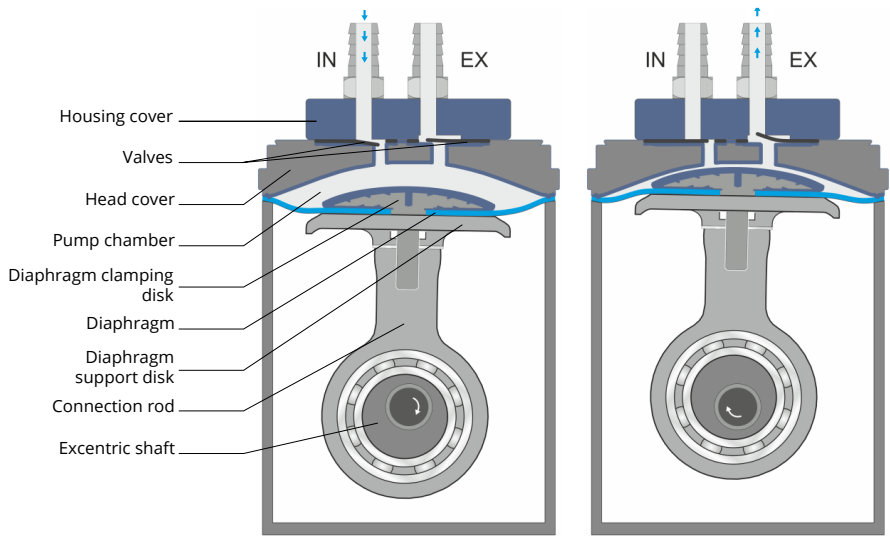
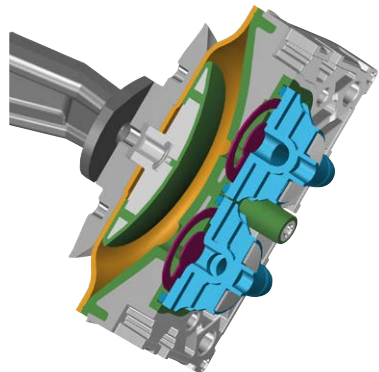


Fig. 4: Schematic representation of a diaphragm pump

It is crucial that the materials used in the pump head are chemical-resistant. Special fluoroplastics which have a high long-term stability and density are used for the manufacture of each of the different parts in the pump head (Fig. 5). Although fluoroplastics are extremely chemically resistant, they are not very mechanically stable; therefore, a metallic stability core inside is extremely important.



- PTFE:** Polytetrafluoroethylene
- ETFE:** Ethylene tetrafluoroethylene
- ECTFE:** Ethylene chlorotrifluoroethylene
- FFKM:** Perfluoroelastomer

(You can find more detailed information on the topic of chemical resistance in our related flyer. You can download it at www.vacuubrand.com in the support/ brochures area)

Fig. 5: Detail view of the pump chamber of a chemistry diaphragm pump

In a rotary vane pump, an eccentrically mounted cylinder with sliding vanes rotates within the cylindrical pump chamber, thus pushing the inflowing gas in the direction of the exhaust. After a certain point in the rotation, the eccentric position leads to the gas being compressed by the movement of the vanes (Fig. 6). As soon as the gas pressure exceeds the opening pressure of the outlet valve, the gas escapes through the exhaust. Oil is used here for lubricating and sealing the vanes to the metal cylinder.

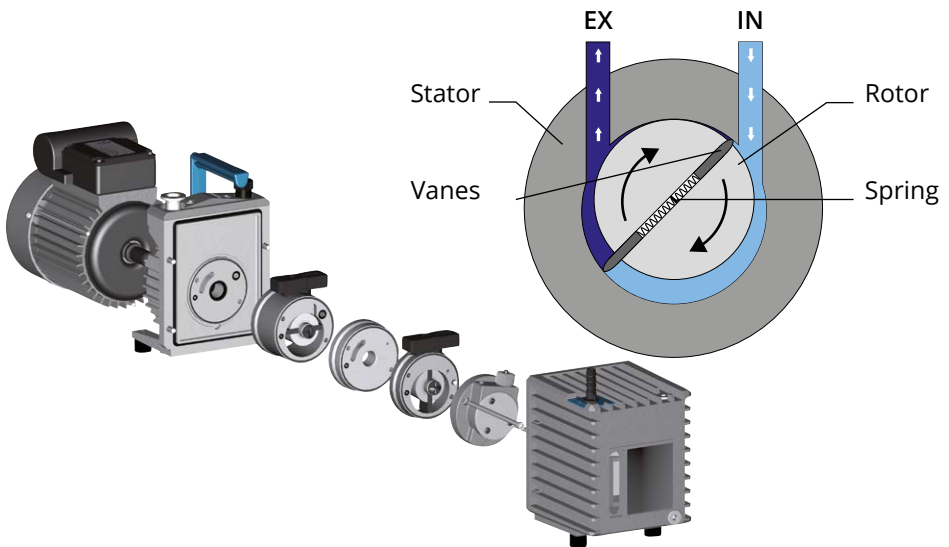


Fig. 6: Construction of a rotary vane pump



Fig. 7: Rotary vane pump RZ 2.5

The advantage of this technology compared to diaphragm pump technology is the deeper ultimate vacuum of up to 10^{-3} mbar with two-stage rotary vane pumps. The disadvantage however is lower chemical resistance, since many parts are made of metal and may corrode when they come into contact with chemicals. In addition, the pumped gases come into contact with the oil. The oil vapours disturb sensitive processes and also the oil is attacked and diluted by these substances. Therefore, oil pumps must be protected from corrosive chemicals and condensation by suitable equipment (see page 12).

In this instance, a hybrid chemistry pump is a very good alternative. This consists of a rotary vane pump combined with a chemical-resistant diaphragm pump that constantly evacuates the oil aggregate during the process, thus extracting the corrosive vapours and condensates from it.

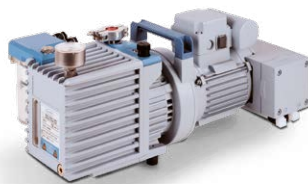


Fig. 8: Hybrid pump RC 6

Single-stage or multi-stage?

The ultimate vacuum and the suction capacity of a pump depend on the modular connection of the pump heads. A parallel connection of the pump heads causes an increase in the pumping speed, while a serial connection leads to a better ultimate vacuum - i.e. a lower ultimate pressure (Fig. 9).

VACUUBRAND uses a serial connection for up to four stages in its diaphragm pumps and attains ultimate vacuums of between 100 mbar (single-stage) and 0.3 mbar (four-stage). In rotary vane pumps, a maximum of two stages are used, which enable an ultimate vacuum in the range of 10^{-3} mbar.

The pumping speed depends on the parallel connections of the pump heads as well as the number of pump heads and the volume of the pump chamber.

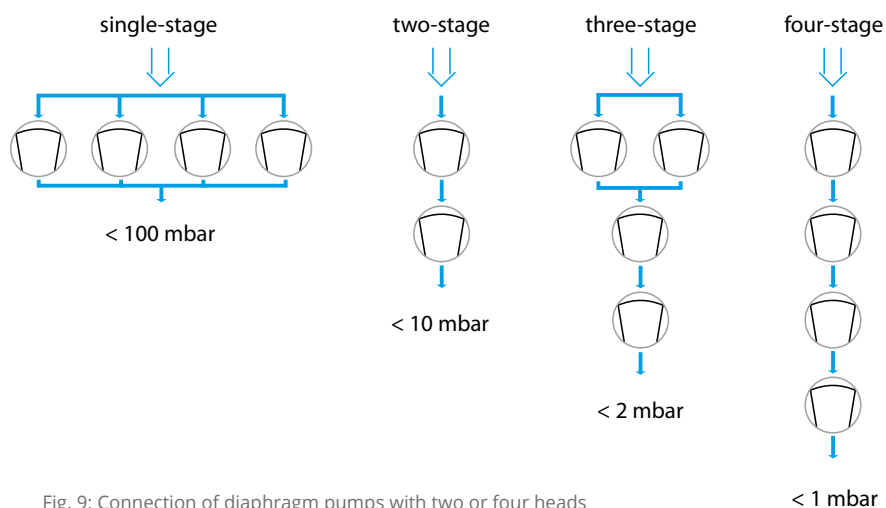


Fig. 9: Connection of diaphragm pumps with two or four heads

Protecting the pump and the environment

Condensation inside the pump can impair its function and leads to premature wear. This problem affects both diaphragm and rotary vane pumps. So many pumps have a so-called gas ballast, which lets in small amounts of air or inert gas through a valve. This minimizes the condensation of gases inside the pump and ensures that already formed droplets are pushed towards the exhaust. For many applications in chemistry this function is indispensable for the protection of the pump. Therefore, a good ultimate vacuum with gas ballast valve open is also very important (Fig. 10). After operation, it is recommended that the pump be left running for about half an hour with the gas ballast open and the pump inlet closed before switching it off. This removes any residual condensates from the pump. Rotary vane pumps should also be warmed up before the start of the process with the valve closed, because a correct operating temperature of the oil contributes to the reduction of condensation build up.

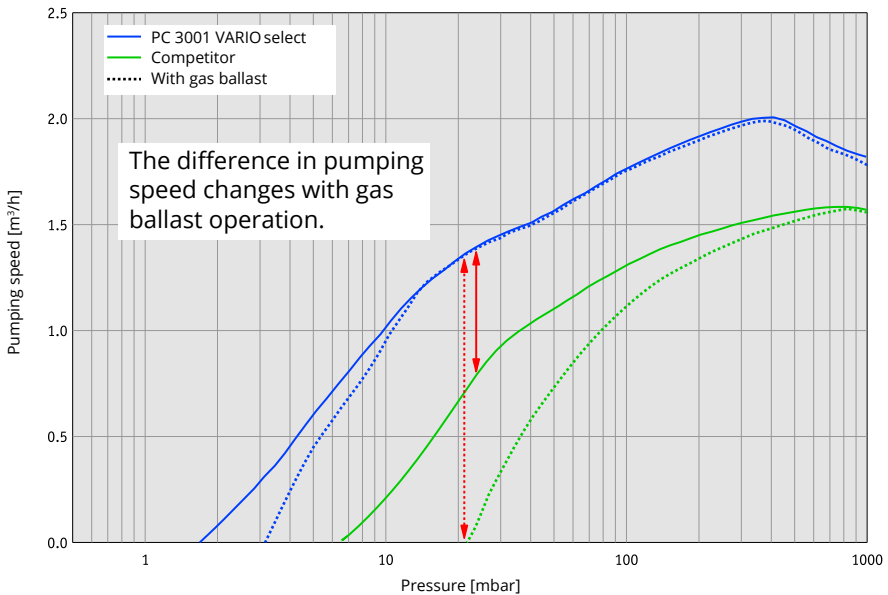


Fig. 10: Comparison of pump curves with and without gas ballast: PC 3001 VARIO select vs. competitor's product

Due to its lower chemical resistance, the insertion of a condensation trap may be necessary in rotary vane pumps in order to capture corrosive chemicals and condensates even before they enter the pump inlet. The use of a chemistry hybrid pump is also useful in this respect, especially in chemistry laboratories where a strong ultimate vacuum is required. To protect the health of the laboratory staff, an oil mist filter is frequently used at the exhaust to prevent the contamination of laboratory and inhalation of oil vapours.



Fig. 11: Rotary vane pump in the pump system version with oil mist filter and cold trap

Modern diaphragm pumping units also have a so-called “condensate separator” at the pump inlet which protects the pump from process condensate droplets and solid particles. An “exhaust emission condensor” at the exhaust side protects the environment from solvent emissions and allows for their recovery. Round bottom flasks catch not only residues of solvents or condensates, but also act at the same time as a silencer, significantly reducing noise emissions.



Fig. 12: PC 3001 VARIOselect pump system with the new vacuum controller VACUU-SELECT. Separator shown at back, and emission condenser shown in foreground.

Measuring vacuum

Depending on the pressure range, different pressure sensors are used for the measurement of vacuum. The rough vacuum range is best served by capacitive sensors. The deflection of a small diaphragm caused by pressure changes is recorded capacitively and converted into a pressure indication. In the laboratory, the use of ceramic diaphragms is useful because these are chemically-resistant and very robust. This principle offers additional advantages such as measurement regardless of the gas type, high precision, low dependency on temperature and good long-term stability. The disadvantage depends on the reduction of the measuring range due to the thickness of the diaphragm.

For this reason, in the fine vacuum range, a Pirani sensor is frequently required. This sensor, named like its inventor, Marcello Pirani, measures the thermal conductivity of the gas related to the respective pressure and can thus determine the existing vacuum precisely. The advantage of this method is the extended measuring range of atmospheric pressure down to 10^{-3} mbar, however, inherent to its functional principle, the best precision is achieved in the range from 10 to 10^{-2} mbar. The disadvantage of the Pirani sensor compared to the ceramic diaphragm is the dependence on the type of gas for the measured results, which, depending on the thermal conductivity of the gas, deviates from the setting for air. In comparison to conventional Pirani sensors, the VACUUBRAND Pirani measuring devices are distinguished by their extraordinary chemical resistance and robustness thanks to their combined plastic and ceramic construction.

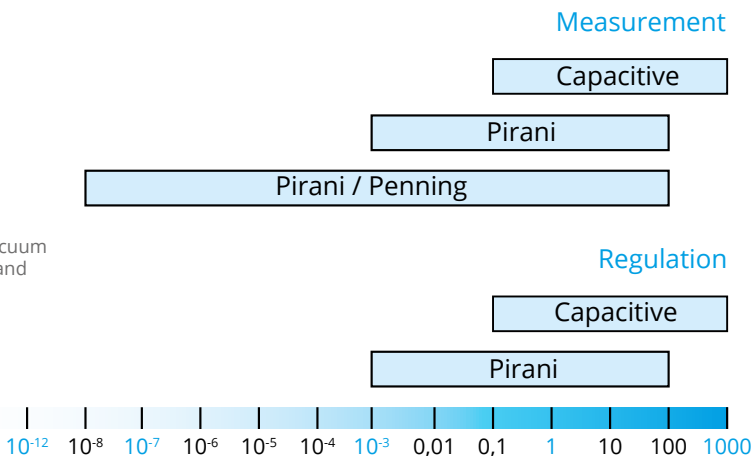


Fig. 13:
Methods for vacuum
measurement and
regulation

If measurements are required to be made across the complete pressure range of rough and fine vacuum, the advantages of both capacitive and Pirani sensor can be used in combination. From the outside, such a combination sensor is not recognizable to the user. Figure 14 shows, for example, the VACUUBRAND combination gauge, which is exceedingly compact.

To verify the required measurement accuracy, customers can have their devices tested and marked in certified laboratories. VACUUBRAND itself operates a DAkkS calibration service that is inspected regularly by the German Accreditation Office.

Regulating a vacuum

The vacuum in a reaction vessel can be regulated in three different ways:

- ✦ by manual alteration of the flow rate
- ✦ by electronic valve switching
- ✦ by rotational speed control

With a simple manual valve alteration of the flow rate, the vacuum can be influenced coarsely without the need for additional aids. However, active and accurate vacuum regulation can only function either electronically via valve switching or via regulation of the rotational motor speed. In valve switching, also often called on-off control, an electromagnetic valve placed in the suction line between the pump and application is opened and closed. This causes the vacuum to fluctuate between two freely definable tolerance values (Fig. 16). Diaphragm pumps with speed control of the motor however, enable infinite adjustment of the suction capacity and can achieve the utmost precision in vacuum regulation (Fig.17). Since, in this case, the pump only runs as fast as necessary, the user obtains huge energy savings (up to 90% in comparison to non-regulated systems). Additionally, wear, noise emissions and vibration are significantly reduced.



Fig. 14: Combination gauge VACUU-VIEW extended



Fig. 15: The new VACUU-SELECT vacuum controller

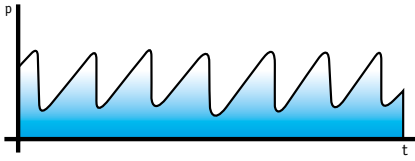


Fig. 16: On-off control through valve switching



Fig. 17: The most precise regulation by speed control

Vacuum regulation which incorporates an automatic function greatly facilitates research work. For this, VACUUBRAND uses the new VACUU·SELECT vacuum controller which in on-off mode uses its detect function to enable automatic boiling pressure determination. The VACUU·SELECT offers even more possibilities in combination with a variable speed pump from VACUUBRAND. This VARIO® regulation not only finds the boiling pressure, but also reacts to changes in it thanks to a unique vapour pressure tracking system. Thus, the vacuum is continuously tracked using vapour pressure and then continually optimised to the process demands. In this manner, the results can be achieved in the shortest possible time with just the push of a button, without the need for monitoring and intervention (Fig. 18).

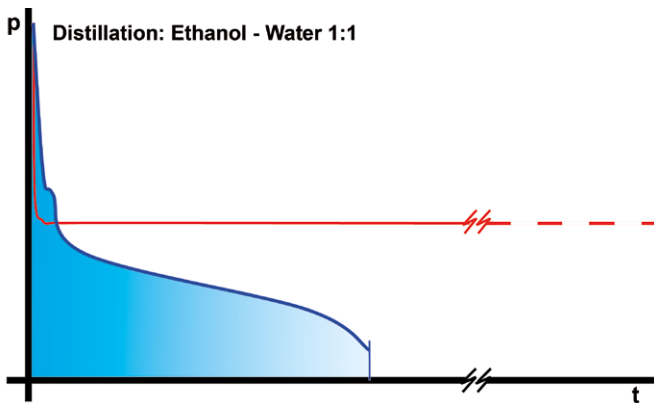


Fig. 18:

- Competitor's product in automatic mode - first steam pressure is maintained, evaporation comes to a standstill because the vacuum is not tracked
- VACUUBRAND VARIO® Control - Complete distillation through adaptive steam pressure regulation in the shortest time

Individual pumps or vacuum networks?

For lab vacuum supply, both individual pumps and multi-user vacuum systems are feasible solutions.

In house vacuum systems, all the vacuum inlets in a building are supplied by a large, centralized vacuum pump. However, house vacuum systems have several disadvantages. Unchecked flow and normal pressure variations within the vacuum piping cause back flow. This back flow causes cross-contamination and disruptive interference among system users. In turn, this can create hazardous mixtures of flammable or explosive gases, or spread infectious materials around the building. Another drawback of house vacuum systems is that pumps are not sized properly to meet actual demand. An undersized pump can lead to insufficient vacuum. An oversized pump results in not only higher construction costs, but also high operating costs and high maintenance costs since central vacuum pumps typically run nearly all the time.

The better modern alternative to avoid those disadvantages is the local vacuum network. Local vacuum networks are a modular solution that is designed to supply lab-scale user groups with vacuum that is deep and stable enough to support even sensitive research work. Local networks can easily be scaled from installations as small as a single lab bench all the way up to cover an entire building with multiple networks. By creating a local network and utilizing

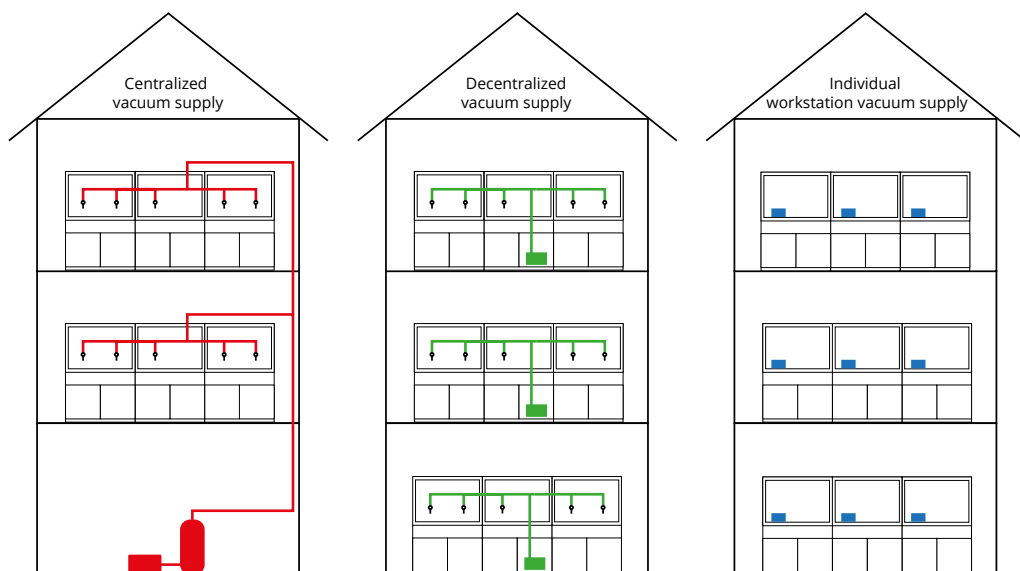


Fig. 19: Different options for vacuum supply

specially-designed vacuum inlets, the risks of cross-contamination and interference are virtually eliminated. And local vacuum networks are an economical choice. Maintenance and operating costs are minimized by using efficient and chemical-resistant diaphragm pumps that are sized for actual demand.

The choice between individual, or point-of-use pumps, and a local vacuum network depends upon several factors. These include the number of points-of-use, the type of applications which require vacuum, and the degree of vacuum control required. Because of the various complexities and subtleties, it is often helpful to take the time to review these and other technical points with an expert.

For lab construction or renovation projects, it is important that the vacuum requirements be considered during the lab programming process. In addition to considering the number of locations which will require vacuum and the type of applications that need to be supported, the chemicals which will be aspirated into the vacuum system should also be considered. This will allow designers to select a properly-sized pump and to utilize tubing and fittings that offer an appropriate degree of chemical resistance.

VACUUBRAND's VACUU-LAN® networks include a wide range of vacuum inlet configurations that include both manual control and precise electronic control. Wetted parts - such as the tubing, fittings, and proprietary fixtures (inlets) - have wetted materials such as PTFE and PVDF to ensure outstanding corrosion resistance against a vast array of chemicals commonly found in laboratories.



Fig. 20: Local network VACUU-LAN®. A pump in the lower cabinet supplies several work stations.

OVERVIEW

A more precise look at the performance characteristics of a vacuum pump and its equipment is always worthwhile. To find the best solution for your laboratory, we have summarized the most important distinguishing characteristics for you once more:

Pump technology (page 8)	Diaphragm pump Rotary vane pump Chemistry hybrid pump
Performance (page 4)	Max. ultimate vacuum Max. ultimate vacuum with open gas ballast Max. pumping speed Sucking capacity curve
Chemical resistance (page 7 + 8)	Corrosion resistance of the materials ATEX authorization
Protection of the pump and the environment (page 12)	Gas ballast Cold trap Oil mist filter Suction-side separator AK Emission condensor EK Chemistry hybrid pump
Measurement technology (page 14)	Rough vacuum: Capacitive diaphragm sensor Fine vacuum: Pirani sensor Rough & fine vacuum: Combination gauge Chemical resistance DAkKS certification
Regulation technology (page 15)	Manual flow rate regulation Valve switching / on-off regulation Speed regulation / mbar exact regulation Automatic boiling pressure detection Automatic vapour pressure tracking
Network solution (page 17)	Chemical resistance of the pipework and modules Prevention of cross contamination



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