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ACHEMASIA '89

Contents

目　录

（中文部分的图、表、参考文献见英文部分。）

VACUUM TECHNOLOGY

FOR

CHEMISTRY

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1. INTRODUCTION

For many decades now, one and two-stage, oil-sealed rotary vane pumps have had an unchallenged position in producing vacuum in chemical laboratories. They have a reputation for being efficient, flexible in what concerns operating pressure and dependable - and this in spite of the fact that vacuum pumps are exposed in chemical laboratories to the hardest conditions imaginable. Thus is seems strange that although their use is increasing, they are not really as popular in the chemical industry as in chemical laboratories. The advantages of oil-sealed mechanical vacuum pumps are obvious:

- A constantly maintained pumping speed down to ultimate pressure (Fig. 1).

- Low ultimate pressures while at the same time compressing against atmosphere (Fig. 1).

- No contaminated operating or waste water.

- Good possibilities of expansion, e.g. by adding Roots pumps to insure high pumping speeds with low ultimate pressures (Fig. 2).

- Easy recovery of pumped off vapours by means of condensation.

2. ROTARY VANE PUMPS IN CHEMICAL LABORATORIES (Fig. 3 + 4)

The following rules should always be followed when using rotary vane pumps in chemical laboratories:

a. One or two-stage vacuum pumps which are to be used in a pressure range above 1 to 5 mbar should always be operated with an open gas ballast valve. If this is not done, an inadmissibly high dilution of the pump oil together with dissolved or condensed vapours can lead to damage of the pump.

b. The pump should not be connected to the vacuum plant until it has reached operating temperature.

c. Before switching off the pump, keep it on running and be sure that the gas ballast valve is open.

d. Operate the pump only with a condenser or cold trap at the suction side (Fig. 5).

e. Avoid liquid shocks and dust.

f. Never block off the pump at the exhaust side.

g. Check and service the pump regularly.

As the application in chemical laboratories has already made clear, the main task of vacuum pumps when used in chemical applications is the pumping off of vapours. This is also the case in a technical laboratory or in chemical production.

3. USING ROTARY VANE PUMPS IN THE CHEMICAL INDUSTRY

As a rule, vacuum processes in chemistry are not used during the reaction step, but rather when reaction products are worked up. In such processes, almost always one takes advantage of the fact that the boiling point of a substance is dependent on the prevailing pressure (Clausius Clapeyron's equatation: $\log p = - A \cdot T^{-1} + B$ (Fig. 6)).

Thus, what is mainly wanted is to transfer substances into the vapour phase under the gentlest conditions possible and at a low temperature. Hence, the most frequent work that a vacuum pump must carry out in a chemical process is the transportation of vapours.

3.1 Pumping Off Vapours

The definition of "vapours" according to DIN 28.400 (Part 1) is:

Vapour is matter in gas form which is either in thermodynamic equilibrium with its liquid or solid phase (saturated vapour) or which at actual temperature can be brought to thermal equilibrium (condensed) (unsaturated vapour).

Contrary to permanent gases, vapours usually have quite a number of unpleasant porperties which must be known if a suitable pump is to be selected or if such a pump is to be adapted optimally to the process conditions. Thus to provide the best possible advice, the consultant must be very familiar with the conditions under which the pump is to be operated.

When planning a vacuum process, the following data, at least, should be taken into account:

- Volume of the vacuum vessel.
- Required operating pressure.
- Desired pump down time.
- Ambient temperature in the pump environment and the gas inlet temperature.
- Type of operation - whether continuous or discontinuous.
- Type and amount of the substances to be pumped off.
- Share of permanent gases or amount of leak air in the system.

In addition, it is important to know any regulations or legal requirements which must be adhered to (emission values, noise or explosion protection).

The question then arises of whether oil-sealed rotary vane pumps are suitable for pumping off vapours. If certain conditions are met, this question can be answered with a clear "yes".

First of all, however, we must recognize the difference between the vapour-suitable and the non-suitable versions of rotary vane pumps.

Difference 1 - Water Vapour Tolerance

This value is the first important point to consider for a good ar less satisfactory vapour tolerance. This value can only be valid for substances not soluble in oil with a similar boiling point to water.

The water vapour tolerance changes as conditions change:

Condition	Effect on Tolerance
High ambient temperature	increases
High temperature in the pump	increases
High counter-pressure	decreases
High amount of permanent gases other than water vapour	increases
Throttling off the gas ballast valve	decreases
Increasing share of vapour in the gas balast	decreases

<u>Difference 2 - High Vapour Tolerance for oil-soluble substances</u>

(As a rule this includes almost all organic solvents)

Even if the vapours running through the pump do not condense, they will dissolve in the oil up to an equilibrium concentration, in accordance with Henry's Law. This can cause considerable dilution of the oil. If the oil is used only for internal sealing and to remove the compression heat, but not used at the same time to lubricate the vane bearings or other sensitive parts, such dilution is harmless. It will simply lead to a balanced situation determined by the amount of gas ballast and by the pump temperature and it must not exceed a maximum admissible value.

Pump manufacutrers do not mention in their catalogues the compatibility of their pumps with oil-soluble substances since this value is not defined in the acceptance rules of PNEUROP. Modern rotary vane pumps permit very high - sometimes even unlimited - partial pressures, for example, for solvent vapours such as methylene-chloride or acetone, at the suction side of the pump.

The following table shows the situation for a one-stage rotary vane pump VAROVAC Type C (Fig. 7) with separate bearing lubrication and a water vapour tolerance of 60 mbar:

Solvent	Vapour Tolerance (mbar)+	Maximum throughput per m^3/h pumping speed (kg/m^3)
Acetone	1013	2.3
Dichlormethane	1013	3.4
Ethanol	266	0.4
Methanol	1013	1.3
Toluene	29	0.1

+ Pump at operating temperature

It is possible to recover the solvents in an emission condenser. Complete systems, therefore, consist of the oil-sealed rotary vane pump as well as of the emission condenser and, under the name SOLVAC, have been successfully used for a very long time now (Fig. 8).

For example, in vacuum drying, oil-sealed rotary vane pumps have considerable advantages in comparison with other systems:

- A pratically constant high pumping speed and high vapour tolerance assure outstanding throughput when pumping off the major part of the solvent at still high pressure (main drying phase).

- Very low residual moistures can be attained. Pumps with a low ultimate pressure are required for the secondary drying phase.

- There is no contamined waste water. The theoretically low emissions from vacuum processes can be realized in practice and with the easiest of means.

3.2 Oils for Oil-Sealed Rotary Vane Vacuum Pumps

The oil in oil-sealed positive displacement vacuum pumps fulfils mainly three functions:

- Internal sealing
- Elimination of the compression heat
- Lubrication of moving parts.

However, the demands made on a pump oil to be used in a chemical process can be relativated as follows:

Through the corresponding design measures such as have been taken in modern vacuum pumps - for example, the separate lubrication of the bearing (Fig. 9) or the intense water cooling - the demands made on the lubricity or on the temperature resistance of the oil can be considerably reduced.

Quite a number of oils with very differing characteristics and properties are available for oil-sealed rotary vane pumps being used in chemistry.

Modern pump designs are such that of the three original functions to be effected by the oil only one still needs to be fulfilled: that of internal sealing. For this reason, the words sealing material is increasingly being used to denote the vacuum pump oil.

The special demands made on the sealing material are very varied in chemical applications. Thus, for example, the following properties are desirable:

- Inert against chemical attack (e.g., resistant to acids, bases, halogens, halogen-catalysed ageing).
- Resistance against strong oxidants such as oxygen, fluorine and chlorine.
- Good solvent properties for problematic, usually organic components of the pumped media such as, for example, oligomers and polymers, resins, crystalline decomposition or reaction products.

The Fig. 10 gives a good idea of the versatility of sealing materials with which modern rotary vane pumps can be operated. It becomes clear that such a broad selection of sealing materials makes it possible to solve many problems that could arise in a chemical application (Fig. 10).

3.3 Example of Application

3.3.1 Vacuum Pump Set made to Order for Chemical Process (Fig. 11)

To permit savings in energy and to decrease pollution of water, steam ejectors in polycondensation plants can be replaced by a mechanical pump system. The main advantages of such a pump system would be lower energy costs and non-contamined drain water.

The suction media are in this case inert gas (nitrogen), alcohols and organic acids. A suction pressure of less than 1.5 mbar and a volume flow rate of approximately 4000 m^3/h were required.

All the required components were installed as compact vacuum units in a frame. The pump set is a three-stage pump set with 2 Roots pumps and an oil-sealed mechanical vacuum pump Type VAROVAC S 400 C (Fig. 11).

Special Design Features

- Roots pumps with gas-tight pistons and inert gas blanketed piston ring seal.

- Oil-sealed mechanical vacuum pump with electric heater (in explosion-protected design) to keep the pump oil warm while the pump is on stand-by.

- Operation with a condenser.

- Automatic flooding of the system with nitrogen at the end of the process.

The above example clearly shows how flexibly oil-sealed mechanical vacuum pumps cn be used today in chemical production processes. In addition, these pumps make an important contribution toward decreasing the emission of pollutants; there is no contaminated water. The emission of solvent vapours can easily minimized with help of a condenser at the pressure side.

3.3.2 Vacuum Pump System for Fuel Oil Distillation

In the following example it was profitable to combine Roots pumps with a liquid ring pump as backing pump.

Light crude results as tops (middle distillate) from a vacuum distillation. In a further step, water, hydrosulphide and other volatile constituents, mostly hydrocarbons, must be extracted to obtain domestic fuel. This is also done under a vacuum that is usually produced by means of steam ejector pump systems. An alternative proposition for such steam ejectors are mechanical vacuum pump systems, provided they are suitably designed to cope with H_2S and hydrocarbons.

Mechanical vacuum pump systems offer outstanding benefits:

- Small quantity of waste water.

- Low energy consumption.

Chemical Vacuum Pump System

for fuel-oil middle distillation/dehydration plant, substituted for a steam ejector pump system.

Operating and Performance Data

Pumped substance	Air	Water vapour	Hydrocarbon vapours	H_2S	Total
Quantity (kg/h)	15	31	125	0.5	171.5

Pumping speed required at 6 mbar: 9800 m^3/h
Pumping speed of pump system at 1 mbar: 12100 m^3/h

Typical Layout Diagram (Fig. 12).

There are good possibilities of decreasing operating costs considerably, e.g. by replacing steam ejector pumps with mechanical vacuum pump systems. The investment costs for these pumps are higher, but such costs are more than compensated by their lower energy consumption, in addition they produce no contaminated waste water.

For these reasons it can be safely assumed that the importance of mechanical vacuum pumps and systems will continue to grow in the chemical industry.

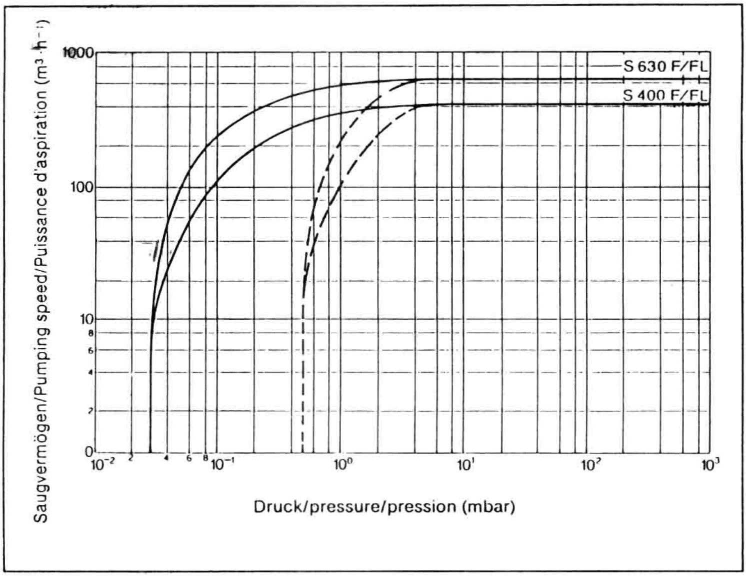

Fig. 1: Pumping speed curves of rotary vane pumps type VAROVAC

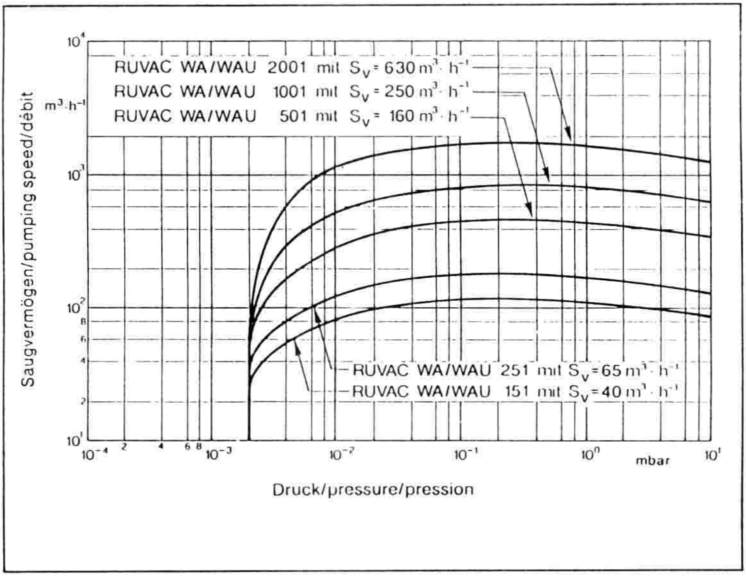

Fig. 2_a: Pumping speed at indicated backing pump speed (example see Fig. 2_b)

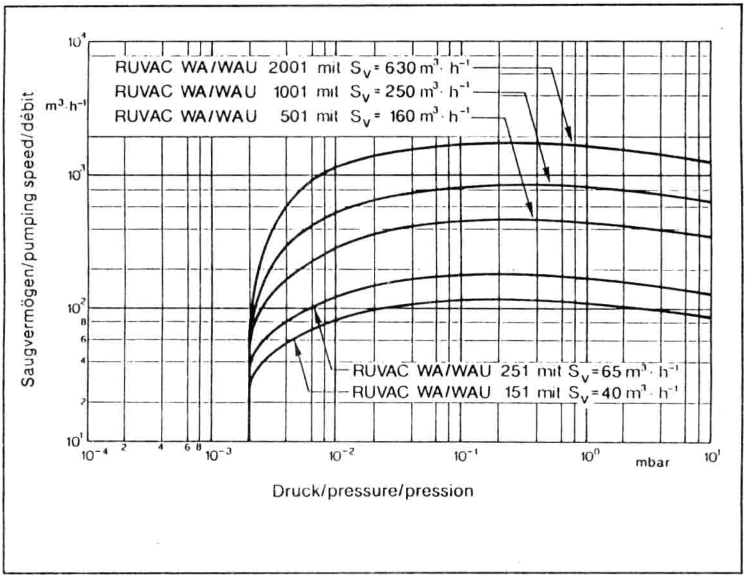

Fig. 2_a: Pumping speed at indicated backing pump speed (example see Fig. 2_b)