

APPLICATION REPORT

Reducing maintenance costs for glove boxes
by using intelligent vacuum systems



Technology for Vacuum Systems



INTRODUCTION

Glove boxes typically use oil-sealed rotary vane pumps to remove air from the main and transfer chambers. However, the maintenance requirements for such pumps are high for safe, continuous operation. VACUUBRAND together with the Inorganic Chemistry and Crystallography Department at the University of Bremen have developed a solution that reduces these costs dramatically.

The Jens Beckmann group at the Institute for Inorganic Chemistry and Crystallography is studying the organometallic chemistry of the light and heavy core elements (www.uni-bremen.de/de/beckmannhtml). The group uses two gloveboxes (Figure 1), both with a large and a small transfer chamber, to work with oxygen- and hydrolysis-sensitive substances. Each glovebox is fitted with a rotary vane pump.

The pressure in the working area of the glovebox must be maintained between +15 mbar and -15 mbar in relation to the atmospheric pressure. If the pressure is too low, the gloves will be sucked inwards and conversely, too much force would be exerted if the pressure is too high. Already when inserting the hands into the gloves and pushing them into the work area, the gas volume in the box decreases and as a result the pressure in the work space increases. This pressure rise is handled by the control of the glovebox opening a valve to the vacuum line of the rotary vane pump until the switching point at the lower pressure range limit is reached.



Figure 1: Glovebox systems at the University of Bremen, equipped with transfer chambers, vacuum pump and vacuum measuring device

THE PROBLEM

In addition to typical organic solvents, salt, nitric acid, aqua regia, bromine, chlorine, ammonia, SO_2 and chlorosilane are also generally used in research with organometallic compounds. Depending on the vapour pressure of the chemicals and solvents, it is possible that some evaporated solvents are evacuated, pass through the glovebox locks and enter the vacuum pump. Once inside the pump these substances can condense and mix in the oil chamber. Some substances form polymer resins with the oil, which make them viscous in the pump. In addition the substances can also cause corrosion inside the vacuum pump.

Dust particles can also get sucked into the pump and reduce the lubricity and anti-corrosive properties of the oil. In addition, this can impact the ultimate vacuum that the vacuum pump can achieve during evacuation of the load lock chamber from atmospheric pressure. The load on the vacuum pump during operation on gloveboxes is therefore composed of four factors:

1. Cyclical evacuation from atmospheric pressure to ultimate vacuum,
2. Compensation of slight overpressure in the glove box,
3. Contamination of the pump and oil by chemicals,
4. Continuous operation, 24 hours a day, 365 days a year.

All in all, these four factors combined led to the required maintenance of the rotary vane pumps at least twice a year. The cost for every maintenance and repair was between 500 and 1000 Euros per vacuum pump. In some cases vacuum pumps were replaced if they were deemed uneconomical to repair. Due to the failure of vacuum pumps members of the work group often could not use the glovebox for extended periods of time. In an extreme case, this prolonged the duration of a PhD study and risked the loss of third-party applications.

The ongoing maintenance, operating and spare parts cost ultimately led Malte Hesse, University Lecturer at the Institute to look for other vacuum technology solutions for their gloveboxes.

The new technology should both reduce the time required for maintenance as well as the cost of operation and spare parts, and increase the reliability of the vacuum supply. An economical solution for the institute was sought in the medium term. This should neither limit the use of the gloveboxes nor make the operation more complicated. The staff using the gloveboxes often changes, therefore simple operation is important.



Figure 2 : Diagram of the pressure area inside the glove box

SOLUTION

VACUUBRAND also produces diaphragm vacuum pumps. The advantage of this technology lies in the chemical resistance of the pumps and in their longevity with minimal requirement for maintenance. However, the achievable ultimate vacuum of 0.6 mbar is not sufficient for operation of gloveboxes if the residual oxygen content must be in the ppm range. The desired oxygen concentration could be achieved with this type of pump only by multiple pumping out of the transfer chamber and flooding with inert gas. This would increase the consumption of argon per sample transfer and considerably extend the time needed for sample transfer.

The chemical resistance of a diaphragm pumps is particularly important in the higher pressure range - for rough vacuum - since the chemical concentration is the highest in this range. The advantage of oil-sealed rotary vane pumps lies in its very good ultimate vacuum of around $2 \cdot 10^{-3}$ mbar, a prerequisite for low residual oxygen concentration during single evacuation. The question now was how to combine the two pumping techniques - oil-sealed rotary vane pumps and diaphragm pumps - in such a way that neither the time for evacuation nor the argon consumption increased. The solution was to use a vacuum controller to automatically switch solenoid valves that connect

the right pumps according to actual process needs. This function allows a valve to be closed as soon as a certain pressure has been reached. The transfer chamber can therefore be evacuated first with a diaphragm pump - like a roughing pump - up to a predetermined pressure and then switched to a rotary vane pump, which then evacuates the chamber to the required ultimate vacuum. This arrangement of dual devices significantly reduces the load on the rotary vane pump since the rough vacuum range, which is not good for the rotary vane pump, can be handled by the more robust diaphragm pump.

A comparison of the pumping curves of a chemistry diaphragm pump and the selected rotary vane pump type showed that the optimum switching point is between 10 mbar and 2 mbar, so that the pumping down time for the transfer chamber does not change significantly (Figure 3).

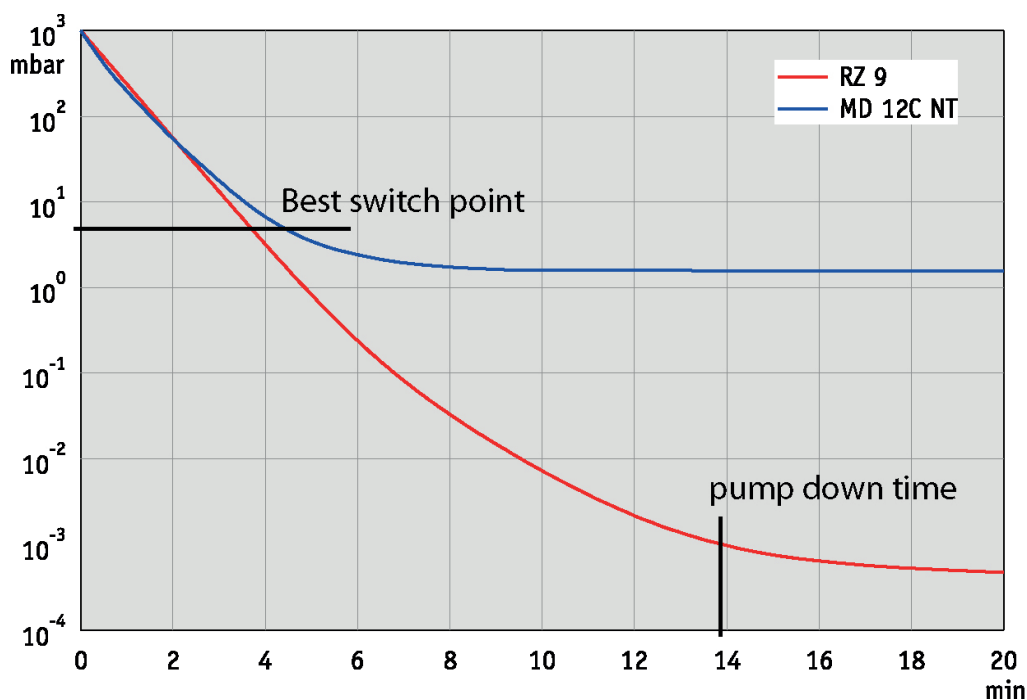


Figure 3: For example by superimposing the pump down curves of a chemistry diaphragm pump MD 12C NT and a rotary vane pump RZ 9 the optimal switching point and the pump down time can be determined.

IMPLEMENTATION

Two rotary vane pumps and two DCP 3000 vacuum controllers with Pirani vacuum sensors were already available at the institute. The institute was initially equipped with a diaphragm vacuum pump, a solenoid valve and a vacuum sensor for the large vacuum chamber. The Department of Domestic Engineering of the University of Bremen installed the pipework.

Joachim Richter, the company's sales representative, and Malte Hesse installed the vacuum technology.

During the six-week test phase, the new vacuum system only ran on one of the glove boxes. Later on this pump combination was expanded for use with both gloveboxes (Figure 4), using one oil pump

for each glovebox but sharing into one diaphragm pump for both gloveboxes. Under the condition that sample transfers is only be done at one of the boxes per time this "double set up" worked perfect.

RESULTS

The system with two gloveboxes, two rotary vane pumps and a single diaphragm pump as a roughing pump has been working constantly in continuous operation for three years now. During this time no maintenance was required on either rotary vane pump. A preventive oil change is only carried out every six months even though the oil doesn't appear different to fresh oil. For the diaphragm pump, a replacement of the wearing parts is now planned, following three years of continuous operation without any need for maintenance. The previous maintenance costs for two rotary vane pumps (approx. € 2,000 to € 4,000 per year) have now been reduced to virtually zero.

The slightly higher investment has already paid off in less than 3 years. The goal of the optimization efforts was fully achieved. The funds saved are available for research work now.

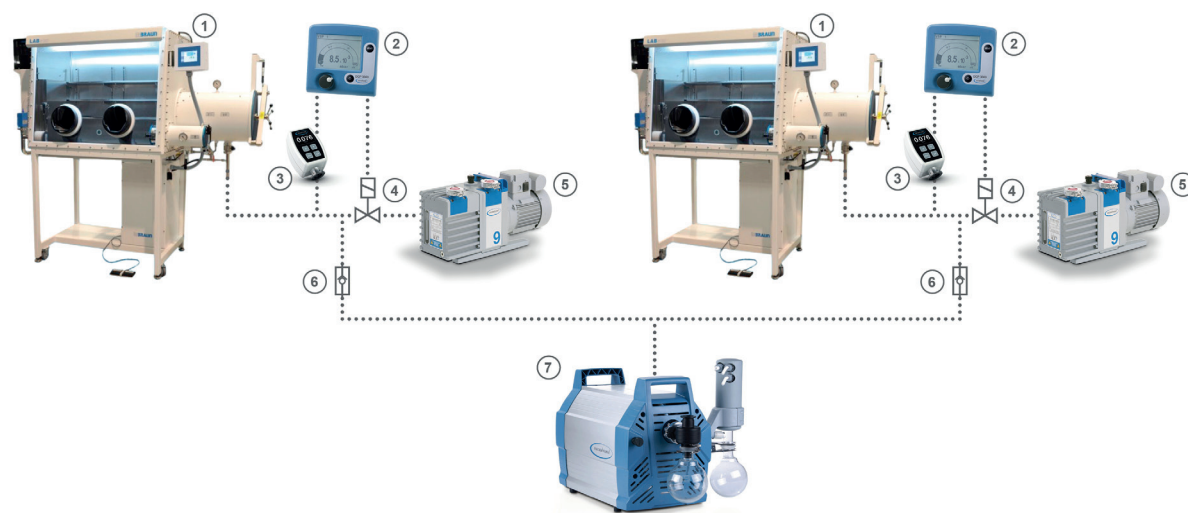


Figure 4: Example of the vacuum supply of two glove boxes

- 1 = glovebox
- 2 = vacuum gauge DCP 3000
- 3 = vacuum gauge VACUU-VIEW extended
- 4 = vacuum valve

- 5 = rotary vane pump RZ 9
- 6 = non-return valve
- 7 = chemistry vacuum system MD 12C NT +AK+EK

Picture: VACUUBRAND, mbraun

Autor: Achim Melching, product manager for VACUUBRAND GMBH + CO KG

2) Dr. Malte Hesse, University lecturer at the Institute for Inorganic Chemistry and Crystallography, University of Bremen

3) Prof. Dr. Beckmann, Professor at the Institute for Inorganic Chemistry and Crystallography, University of Bremen

4) Joachim Richter, Sales Office North for VACUUBRAND GMBH + CO KG