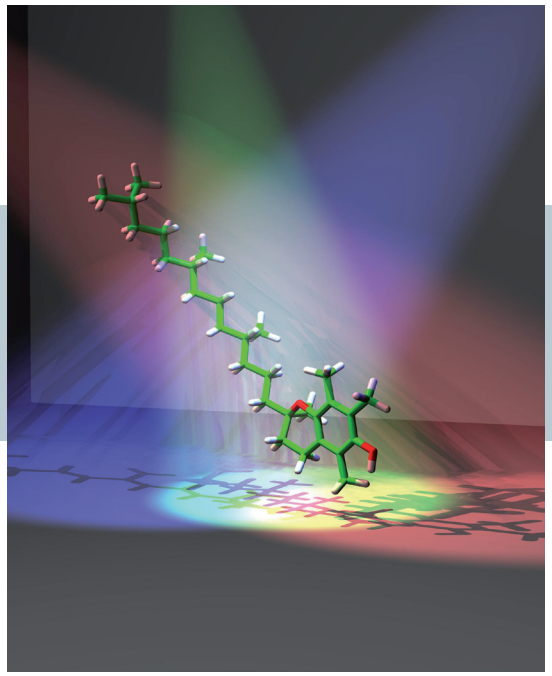
Process optimization in Miniplants

The miniplant facilities at DSM are constantly updated to the latest production technology. In particular, the vacuum supply has been changed successively in recent years. Significant reductions in leakage and precise vacuum control have reduced costs and downtime.

Achim Melching, Product Manager, VACUUBRAND
Dr. Jürgen Dirscherl, Technical Director, VACUUBRAND



Vitamin E-Structure

DSM Nutritional Products is one of the leading manufacturers of vitamins, carotenoids, custom nutrient blends and other additives for manufacturers of feed, food, beverages, dietary supplements and personal care products.

Most of the active substances of DSM cannot be used as ingredients in their pure form. The task of product-form development is to convert them into formulations intended for commercial use, such as additives for food (ice cream, drinks), pharmaceutical products (tablets, capsules), feed (cube, premixes) or cosmetics.

Always the latest technology

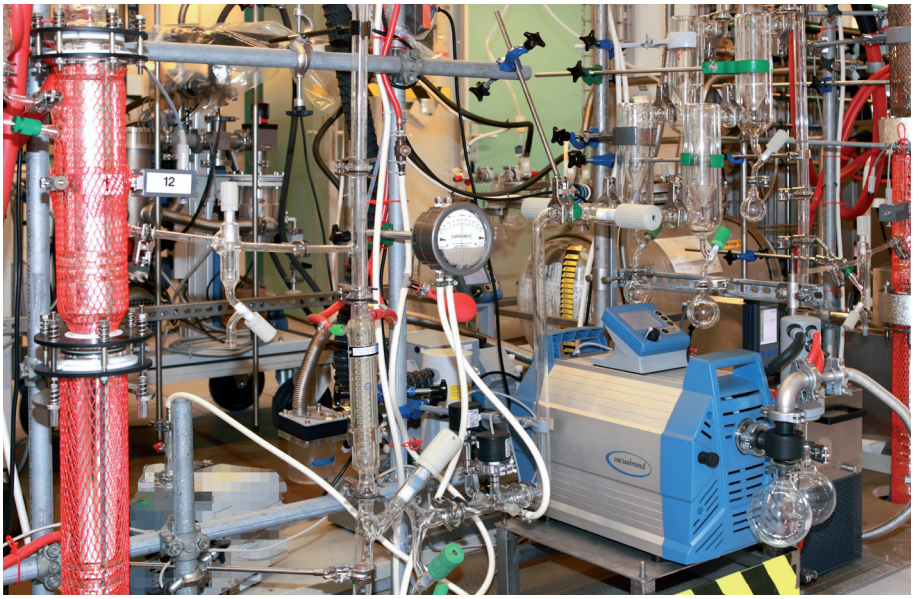
The development of new product forms begins in the laboratory in research laboratories in Kaiseraugst in Switzerland. Bioavailability, stability and solubility are the most important characteristics to be considered at this stage. The focus is on new technologies and new materials. The laboratory development work is supported by a miniplant technical center. Scale-up processes are carried out in miniplant facilities in Sisseln, Switzerland, where processes are developed, optimized and evaluated in order to apply them to the production. The pilot plant, operating in parallel with the scale-up program, supplies larger amounts of product for application and efficacy testing by customers and for further R & D testing.The miniplant facilities are constantly modernized with the latest production technology. In particular, the vacuum supply has been changed gradually in recent years. The oil-lubricated rotary vane pumps, which have been used routinely until few years ago, have been replaced with modern, dry-running, mostly speed-controlled chemical-resistant diaphragm pumps for many chemical processes. Rotary vane pumps are now only used for high vacuum processes such as short path distillations.

Diaphragm pump beats rotary vane pump

The possibility of replacing the rotary vane pumps, at least partially through diaphragm pumps seemed pretty remote at first, as the rotary vane pumps provide higher pumping speed and reach an ultimate vacuum of much greater than 1 mbar. Chemistry diaphragm pumps have the advantages of being free of oil and chemically inert, but can only reach about 1 mbar ultimate vacuum.

To determine if the replacement was possible, the technicians in charge had to analyze more precisely the vacuum requirements of the processes:

- The operating vacuum of most applications is in the range from 500 mbar to approximately 1 mbar, and is therefore accessible with diaphragm pumps.
- The required pumping speed is expected to be nearly zero once the process reaches the stable phase, that



Miniplant set-up with new 8-cylinder diaphragm chemistry pumping unit PC 3012 NT VARIO

is, after the evacuation to the operating pressure. Since most process vapors are condensed before reaching the pump, it is simply a matter of compensating for system leakages and the small amounts of steam not yet condensed to have sufficient pumping speeds to maintain the operating vacuum.

Previously, the process pressure was controlled by solenoid valves, either by metering in small amounts of air (or nitrogen), or by alternately opening and closing the intake line as needed to maintain target operating pressures. The vacuum pump was running constantly at full speed in each case. The "two-point control" provided by solenoid valves regulates the vacuum by managing the hysteresis around the optimum process pressure, though at some cost to efficiency and solvent recovery.
When working with the oil-sealed rotary vane pumps, the sometimes harsh chemicals or solvent vapors had to be condensed or frozen out by cold traps to protect the pumps. Nevertheless, some of the fumes always reached the rotary vane pumps, so the oil in the pumps had to be changed 1 to 2 times per month on average – in extreme cases, as often as weekly. Occasionally, a complete rebuild of the pump in the company's workshop or by the manufacturer was required. During such service interruptions, the pumps were not available, and so a corresponding number of standby pumps had to be available for substitution.

Potential for optimizing the installation

Further potential for optimization of the miniplant derives from the nature of the equipment used. The processes are usually multi-step operations such as rectifications, distillations, evaporations, syntheses or reactions and recrystallizations. Solvent mixtures must be separated, purified and the energy balance determined. For those purposes, equipment such as distillation columns with diameters up to 70 mm and a height up to 14 m are used. Similarly, rotary evaporators with flask sizes of up to 50 l, and reactors in the range of 0.5 to 25 l volume are used, along with suction filters and extraction devices. The plants consist mainly of glass components. There are around ten process set-ups, each of which is adapted to the specific process requirements as needed.

Leaks have been found to occur at the joints between the various glass components. The leaks are extremely undesirable, since they increase pumping requirements and lead to unnecessarily large and therefore inefficient vacuum pumps, and also because ambient air can have adverse impact on the reproducibility of the process.

Historically, the joints between the glass components were usually sealed with PTFE inserts. PTFE offers the advantage of excellent chemical resistance, but has the drawback that it can't adapt adequately to the fine surface structures of the ground glass joints. The result has been considerable leakage. The search for alternative connection methods with significantly reduced leakage

led to seals made of the perfluoroelastomer FFKM to replace the PTFE gaskets.

Savings with FFKM seals

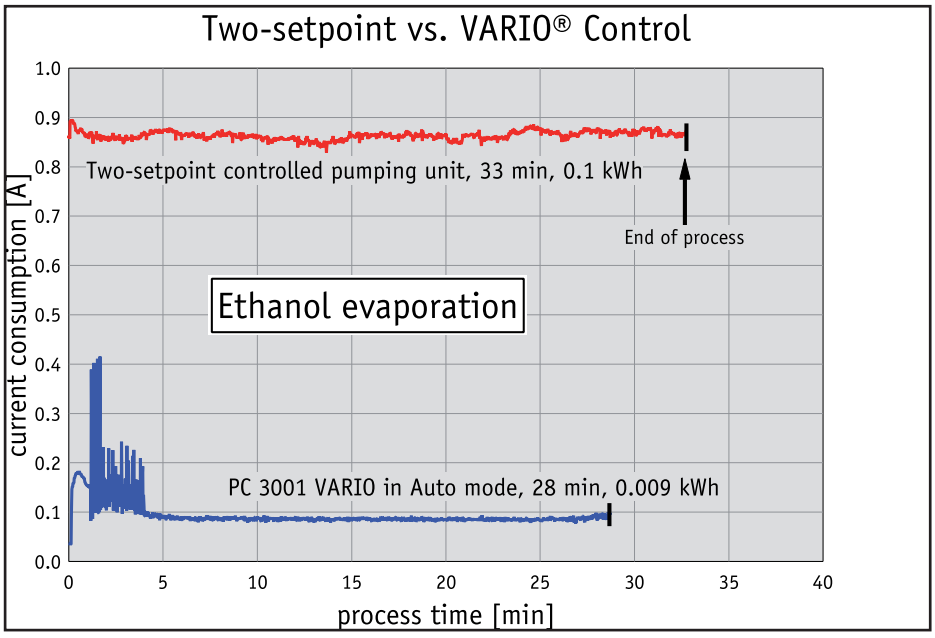
In order to avoid the damaging leaks, there was no need to rebuild the plant completely; the PTFE seals simply were replaced with FFKM. The high price of the FFKM material compared with PTFE is offset by the benefits of the greatly reduced leakage, and a chemical resistance similar to PTFE. In addition, the FFKM seals are also significantly less sensitive to mechanical damage and thus much more convenient to work with in regular use.

For example, it normally takes about two man-months' work to assemble a column with an active height of 14 m. By using FFKM seals, at least one or two weeks can be saved in building the sealed system. The rate of increase in pressure during the leak tests of a set-up equipped with FFKM seals is approximately 0.5 mbar/h which corresponds to a leak rate in the range of 0.01 mbar * l/s. During the leak tests, ultimate vacuums in the range of 1×10^{-2} mbar can be achieved with rotary vane pumps – well beyond the range needed for the process. Due to the greatly reduced leakage with the new seals, chemical-resistant diaphragm vacuum pumps with variable speed control can now be used in kilolab/miniplant facilities, as they have been used for years with great success in chemical laboratories.

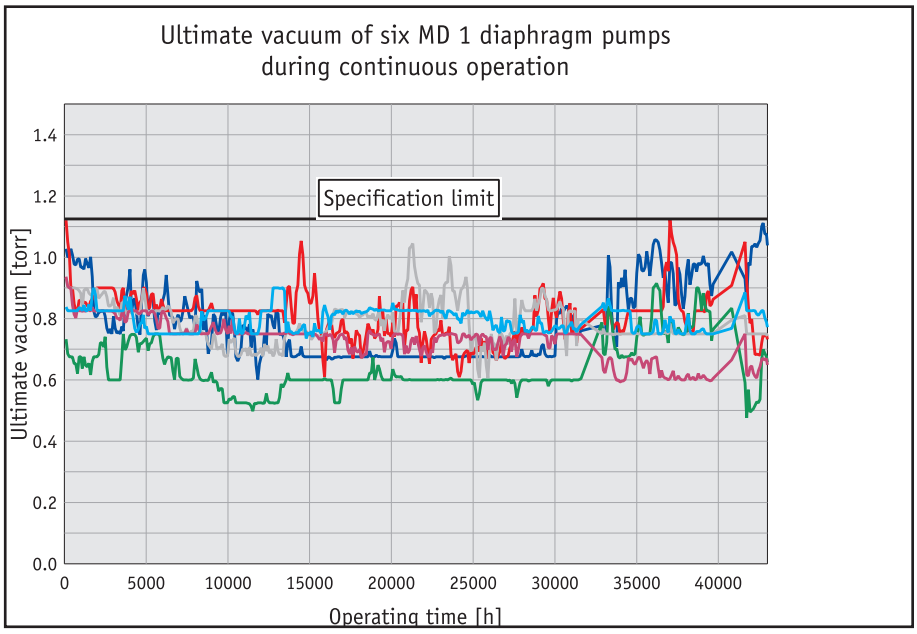
VACUUBRAND chemistry diaphragm vacuum pumps are highly resistant to aggressive chemicals and thus require much less maintenance in such applications.

This reduces:

- operating costs: because of the variable speed control in the process, the pump speed is reduced (often near zero) and saves as much as 90% of the driving energy;
- downtime: as oil changes are eliminated and maintenance intervals of diaphragm pumps are extremely long;
- costs for disposal of used oil are completely eliminated because the diaphragm pumps run dry and free of oil;
- purchase and cost of spare parts for solenoid valves, because the speed-controlled diaphragm pumps precisely control the vacuum to the desired set point, keeping it constant without any hysteresis;
- the complexity of the system by eliminating solenoid valves, chokes and supplemental air terminals. The number of connections and, thus, sealing points also decreases; and,
- staff costs for the maintenance of rotary vane pumps and valves.



Power Consumption VARIO®-control vs. two-point control
- Up to 90% energy savings



Reliability testing
ultimate vacuum of 6 MD 1 diaphragm pumps during continuous operation
- 5 years without maintenance

Increasing efficiency with VARIO® control

In addition to these cost considerations, process reproducibility is significantly improved by reducing leaks and by the accurate and precise vacuum control provided by the variable speed pumps. Chemistry-diaphragm vacuum pumps are directly connected to the process control systems via VACUUBRAND's CVC 3000 vacuum controller, and are thus fully integrated into the control and data acquisition systems. The aim of the miniplant facility, among other things, is to produce accurate process data, on which the process optimization and scale-up can be based. In addition to the controllers, DCP 3000 vacuum gauges can be used with a capacitive sensor (VSK 3000) to detect the pressures locally at various parts of the setup. Of course, these data are also collected centrally via the interface.

The new eight-cylinder pump family from VACUUBRAND, with significantly increased pumping speeds – up to 20 m³/h – and provide ultimate vacuums of 70 to 0.6 mbar, extends the field of application of this modern technology considerably. The high pumping speeds offer significant benefits for miniplant and kilolab use. Especially the substantial improvement of the ultimate vacuum even with gas ballast is beneficial for processes with condensing media.

In the kilolabs and miniplants, the benefits provided by VARIO® variable speed pumps – precise vacuum control, fully functional data interface, low power consumption, low maintenance – are particularly valuable. This technology supports the ongoing modernization of the miniplant facilities at DSM, and provides an excellent foundation for further savings and efficiency gains in the future.

Decision-Facts for users

- **Effective control of system leaks made the use of chemical-resistant diaphragm vacuum pumps with variable speed control possible at DSM.**
- **The chemistry diaphragm vacuum pumps are highly resistant to aggressive chemicals and thus much less maintenance intensive than rotary vane pumps**
- **Dry operation and variable speed pumping reduces operating costs, maintenance costs, downtime, cost of waste oil disposal, acquisition and replacement costs for solenoid valves, and personnel costs.**
- **In addition to these cost considerations, the process reproducibility is significantly improved by reducing leaks and by accurate and precise vacuum control.**