Innovative Vacuum Regulation for Rotary Evaporators

The different methods of vacuum regulation in rotary evaporators are compared by their benefit to the user. Essential criteria of such analysis are process time, rate of solvent recovery, ease of handling and flexibility. Speed controlled diaphragm pumps with automatic regulation have obvious advantages as well as an extremely long life of wear parts.

Vacuum as a tool in the chemistry laboratory

Over the years, the diaphragm pump has become a standard item in a chemistry laboratory, with a variety of applications now featuring regulated vacuum pressure. A typical example is the rotary evaporator, which uses adequate pressure to get as close as possible to a solvent's boil point and to evaporate solvent at low temperatures (Figure 1). If the pressure is too high, evaporation will take too long, while if it is too low, the substance may start foaming. The question is how best to regulate pressure, either manually or automatically? If automatically, how can it be done? Methods of regulation are essential for all aspects of performance, economy and environment. Today, it is unheard of to adjust hot bath temperatures by hand. A skilled user with time on their hands could readjust using a rotary knob, which would provide the same quality as an electronic control circuit but is a rare occurrence in the modern labs of today. Very few people today use a mercury thermometer but instead use an electronic control with PID characteristics.

With patience, the manual system will reveal proper results but most users don't want to monitor best pressure over long time periods. To address this, we need to imagine that a hand-regulating valve doesn't adjust pressure but adjusts suction power instead. During evaporation, temperature and solvent composition are going to change continuously, hence, boil pressure is going to drift and the user will need to constantly readjust the valve by hand. In today's science, with its high costs and time pressure, this process is inadequate.

As a solution, we need to focus on the development of automatic vacuum regulators that at least maintain their pressure level once set. This is currently viewed as state-of-the-art and a variety of approaches have been made. A conventional method is flow regulation by a two-point solenoid valve, whereby the pump runs at full power all the time and as soon as actual pressure exceeds a selected nominal value (nominal plus hysteresis), the valve opens and the system is evacuated until nominal pressure drops below nominal. Such systems used with appropriate knowledge and care will deliver acceptable results. Simple regulators involve problems with pressure 'undershoot' or 'flutter', which may cause foaming or boiling over of the solvent.

Another problem is that such systems with their typical high hysteresis are going to work above their optimum operating pressure most of the time. Low efficiency goes hand-in-hand with long evaporation times. Furthermore, most of the usual vacuum regulators do not really work automatically and the user needs to know and select both adequate nominal pressure and hysteresis. Unfortunately, such values are different for every solvent mix and temperature.

Once evaporation has started, the temperature in the evaporating flask will be determined by the heating power of the hot bath, the evaporation heat of the solvent and therefore, the quantity of the solvent. Some manufacturers try to compensate for this fact with pressure steps or ramps. This, in turn, will increase the number of vacant parameters that need to be programmed into the controller. How should the user know about the optimum pressure curve? These systems are unable to detect and control boil pressure levels automatically. Such control action would not be reasonable with a 'coarse' method of vacuum regulation that does not use more than one single open/close valve.

Speed-controlled vacuum pump systems with a fully automatic controller

This approach features continuous vacuum regulation via a speed-controlled motor driving a diaphragm pump. This concept is simple but the economic realisation of a frequency control requires detailed technical knowledge. Last but not least, the integration of a PID-controller will solve many problems, with undershoot, overshoot and other similar deviations becoming a thing of the past. (Figure 2).

Evaporation and drying are intricate processes that are influenced by a variety of parameters. Simple sequencers for speed-controlled diaphragm pumps show hardly any advantages over open/close valves, since they also depend on the user programming in nominal pressures. Fully automatic regulators detect the boil point (based on vapour temperature) or boil points of various substances in the solvent and therefore approach these points softly without any overshoot and follow-up temperature changes in the hot bath. The regulator exactly controls pressure as required by the specific boil point to achieve as short as a process as possible.

A comparison of open/close valves against fully automatic speed control

The principle of continuous vacuum regulation is shown in Figure 3. Instead of typical flutter of actual pressure around nominal, as is typical for an open/close valve control, a continuous approach to and follow-up of boil pressure is reached.

Figure 4 shows the time saved by using a speed-controlled system with boil-pressure follow-up (LABOROTA 4003 control Mode T auto), in comparison with a conventional open/close valve control without boil-pressure follow-up. The test conditions were identical and the results show that distillation is faster when using a speed-controlled system with boil-pressure follow-up. This time saving will cut down both process times and cost. Integrating daily labour costs while using conventional pump stations in a 'cost of ownership' calculation, mean speed-controlled systems with boil-point follow-up will be far ahead.

The speed-controlled system features distillation in equilibrium with optimum efficiency. Solvent recovery rate is close to 100%, a value difficult to achieve with a simple manual regulation or programmed pressure steps. With open/close valve systems, a significant quantity of solvent gets into the pump when undershooting nominal pressure, which in turn will cause condensation problems in the housing and valves. In particular, two-cylinder diaphragm pumps are prone to condensation and need gas ballast, which in turn affects final pressure. The only solution is a three-stage pump, preferably with speed control.

In addition to its extraordinary regulating properties, a speed-controlled pump features even more benefits for the user. The pump will run only when needed, ie., at minimum speed of a few Hz, which means low noise, low vibration and extended performance life of both diaphragm and valves. The performance life of a diaphragm is expressed in strokes. A 10,000-hour performance life of the diaphragm (at 50 Hz) will mean many years of trouble-free operation of VACUUBRAND diaphragm pumps in speed-controlled systems.

Figure 1 A rotary evaporator, speed-controlled vacuum pump and re-circulating cooler as a modular system.

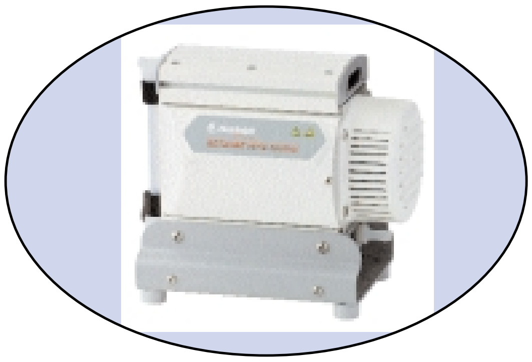

Figure 2 ROTOVAC vario control — speed-controlled diaphragm pump for Heidolph rotary evaporators.

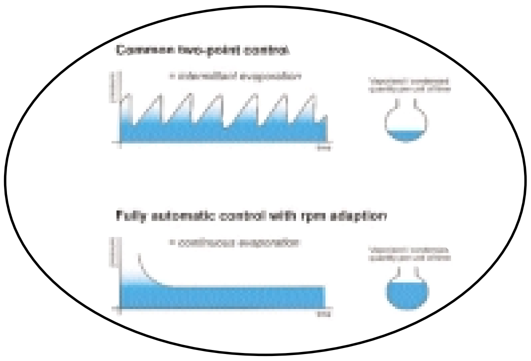

Figure 3 Schematic pressure curves for open/close valve control and the VARIO systems.

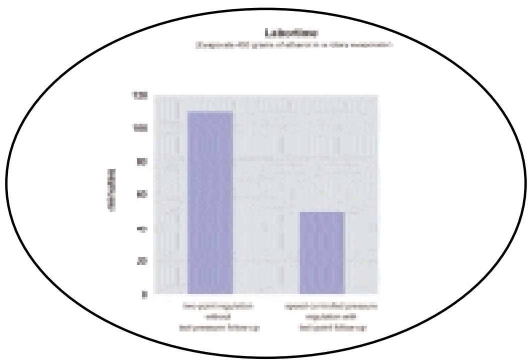

Figure 4 Time taken to evaporate a solvent in open/close valve and speed-controlled systems.