Banishing the mysteries of evaporation and concentration

The concluding part of this two-part article by Dr Induka Abeysena and Rob Darrington of Genevac Ltd describes the latest developments in pumps, cold traps and condensers used in performing the various evaporation and concentration techniques described in Part 1.

In writing this article our aim has been to enhance the understanding of the processes of evaporation and concentration and how they work at a practical level, and through this to enable users to truly optimise their solvent removal processes. In Part 1 of the article (***sp²***, April 2006, pages 48-49) we reviewed the principles and methods of evaporation and concentration.

In Part 2 we review the wide variety of hardware used in evaporation and concentration including pumps, cold traps and condensers. This article also reviews the benefits of a cold trap, the effects of pressure control and how it can be applied to speed up evaporation and prevent sample loss due to sublimation.

Vacuum pumps

Vacuum evaporation systems, such as profiled in Part 1 of this article, require a vacuum source, normally a vacuum pump. Traditional vacuum pumps have used mineral oil for the lubrication of the pump vanes. While such pumps offer good performance (eg below 0.2 mbar (0.07 Torr) with a high flow rate), the mineral oil is both messy and, over time, can be degraded by solvent vapours, causing loss of performance, and in extreme cases pump seizure.

In some laboratories, tap aspirator pumps are still used to create a vacuum. However, the high water usage of such systems and the negative environmental impact of solvent vapour condensing in and contaminating the water has led to their decreased usage.

Fig 1. An oil-free scroll pump as used in the Genevac EZ-2 Centrifugal Evaporator.

The introduction of a new generation of dry (oil-free) pumps (Fig 1) for use with evaporators and concentrators combines high performance (0.2 mbar at flow rates of 80 l/min and more) with very low maintenance and minimal environmental impact.

Cold traps and condensers

In a concentration or evaporation system a cold trap or a condenser functions as a solvent recovery system. Although it was first used to protect sensitive vane pumps from solvent attack, a well-designed cold trap can also be advantageously used to speed up the evaporation process. When solvents vaporise there is a huge volume expansion, something of the order of 20,000 times, and when the cold trap condenses vapours back to liquid, the corresponding volume reduction helps to pull a vacuum and speeds up the concentration process considerably.

Traditionally, cold traps have been made in the form of a stainless steel vessel with cooling coils attached to the outside. Typically the vessel is connected in the vapour path between the concentrator and the pump. The vessel is chilled to below 0°C by a gas compressor system, similar to that used in a refrigerator. A common problem with this design is the difficulty and time lost in emptying condensed solvent. To empty a traditional cold trap, especially when water is the solvent being condensed (as this freezes to form ice), the cold trap has to be defrosted before the trap can be emptied. To overcome this, some suppliers have introduced an interchangeable glass flask, which sits in the cold trap and collects the solvents. At the end of the concentration process the flask is exchanged for a fresh flask. However, to employ this method a thermal transfer fluid (normally silicon-based) is used to bridge the thermal gap between the stainless steel vessel and the glass flask. Changing cold flasks covered in very slippery fluid is potentially dangerous, thus the method has not proved popular.

While limited in terms of sample throughput, rotary evaporators overcome both these issues by collecting solvent as a liquid in a glass flask. The simplest glass condensers operate by chilling the outside of the glass with cooling water or dry ice.

The very latest gas compressor cold traps, such as the miVac Speed Trap shown in Fig 2, work in a similar way to a conventional Leibig condenser, in that the solvents are collected directly into a glass vessel on the front of the trap. The cooling coils are suspended directly in the vapour path and deliver up to 50 per cent more condensing power than traditional cold traps, providing higher solvent recovery. The miVac Speed Trap design also requires no cooling water or dry ice to operate and the

glass flask can be easily removed with a single quarter-turn action.

When selecting a cold trap, the condensing power is more important than low trap operating temperatures. For example, some commercial cold traps run at low temperatures eg -80°C or -104°C, but they perform inefficiently, as they consume almost all available power to reach the low temperature rather than help condense solvent vapours. Gas compressors provide cold traps with full condensing power down to about -20°C. Figure 3 illustrates the simple physical law governing any gas compressor used as a chiller. To gain optimal performance from a cold trap, it is critical to operate with full condensing power, and in the case of a gas compressor system this means controlling the boiling point of the solvents to -20°C or above. In a vacuum system this requires a pressure controller and knowledge of the solvents used.

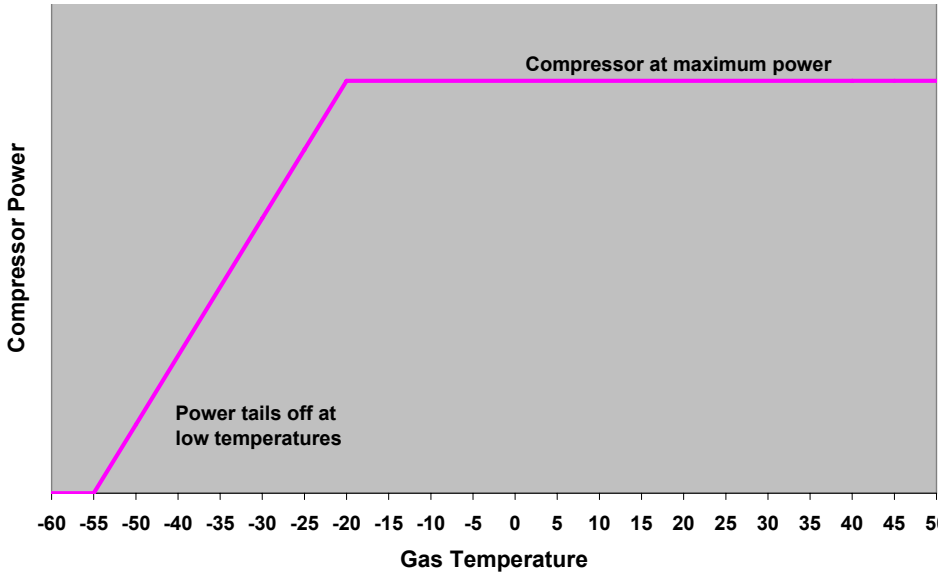

Fig 3. Cold trap power tails off at lower temperatures.

The effect of pressure control

Pressure control in a vacuum evaporation system is critical for a number of reasons: to ensure optimum trapping of evolved vapours; to speed up the evaporation of complex mixtures; and also to prevent sample loss by sublimation.

Regulating solvent boiling temperature in a vacuum concentrator is typically done by controlling the pressure (see principles of evaporation in Part I of this article). By controlling the boiling point, and thus the vacuum concentrator condensing point to -20°C, solvent is condensed at the temperature where the gas compressor cold trap has the most power, and is therefore most efficient. (It is possible to operate at higher temperatures, however this would slow down the heat energy input to the samples and the overall process. Additionally, most users prefer their samples cold, eg methanol can be efficiently boiled off and condensed at 11 mbar pressure at -20°C). Some solvents freeze at low pressures, which may be undesirable in a concentrator, therefore the pressure needs to be kept higher if this is the case. Genevac's research has found that the optimum pressure for water concentration is 8mbar, at which pressure water boils at +4°C. In the case of complex mixtures, HPLC fractions for example, where water and an organic solvent are present, the organic solvent must be removed without freezing the water, or evaporation is very slow. A specific technical note on this application is available from Genevac, see the contact details below.

Most samples can become volatile under the right conditions. Generally the smaller the size of a molecule the easier it is to volatilise, and this is especially true for organic molecules. However, when a sample is of low molecular weight (< MW 300) and/or has high volatility, for example a straight-chain organic molecule with few side-groups, then some sample may also be lost through sublimation during the evaporation process. A detailed technical paper demonstrating the importance of stopping the evaporation process as soon as the samples are dry is available from Genevac.

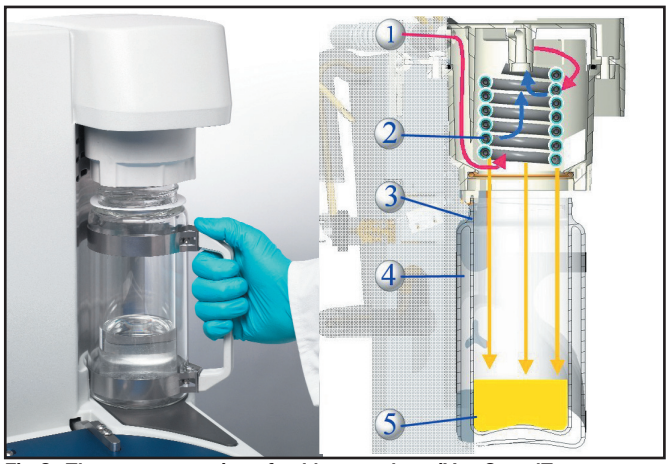

Fig 2. The new generation of cold trap - the miVac SpeedTrap. Legend: 1. Hot vapours enter. 2. Condensing coils with ice shell. 3. Glass collecting flask. 4. Vacuum insulation. 5. Solvent collects as a liquid.

To summarise

The correct choice of vacuum pump and cold trap is critical to ensuring optimum evaporation and concentration performance. Pumps with appropriate vacuum level and having high flow rates are recommended. Highly efficient cold traps are available that not only speed concentration and drying rates, but that also do not freeze recovered solvents and therefore eliminate time lost during the defrosting period. For those working with samples of low molecular weight, pressure must be controlled to prevent potential sample loss due to sublimation. sp²

FURTHER INFORMATION

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