White Paper

OPTIMIZING SOLVENT EVAPORATION PROCESSES

WELCH
An Ingersoll Rand Business

Fast and Accurate Automated Vacuum Solutions for Rotovap Applications

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Rotary evaporation is a laboratory technique commonly used for a variety of applications such as the removal (distilling or “stripping”) of a solvent or solvents from a sample, concentration of a solution, recycle solvents, or synthesize chemicals. This highly useful process is carried out with the help of an apparatus called rotary evaporator and is utilized in a number of fields including university organic chemistry, biotechnology, pharmaceutical and food chemistry.
The apparatus can range in size from bench scale to pilot scale.

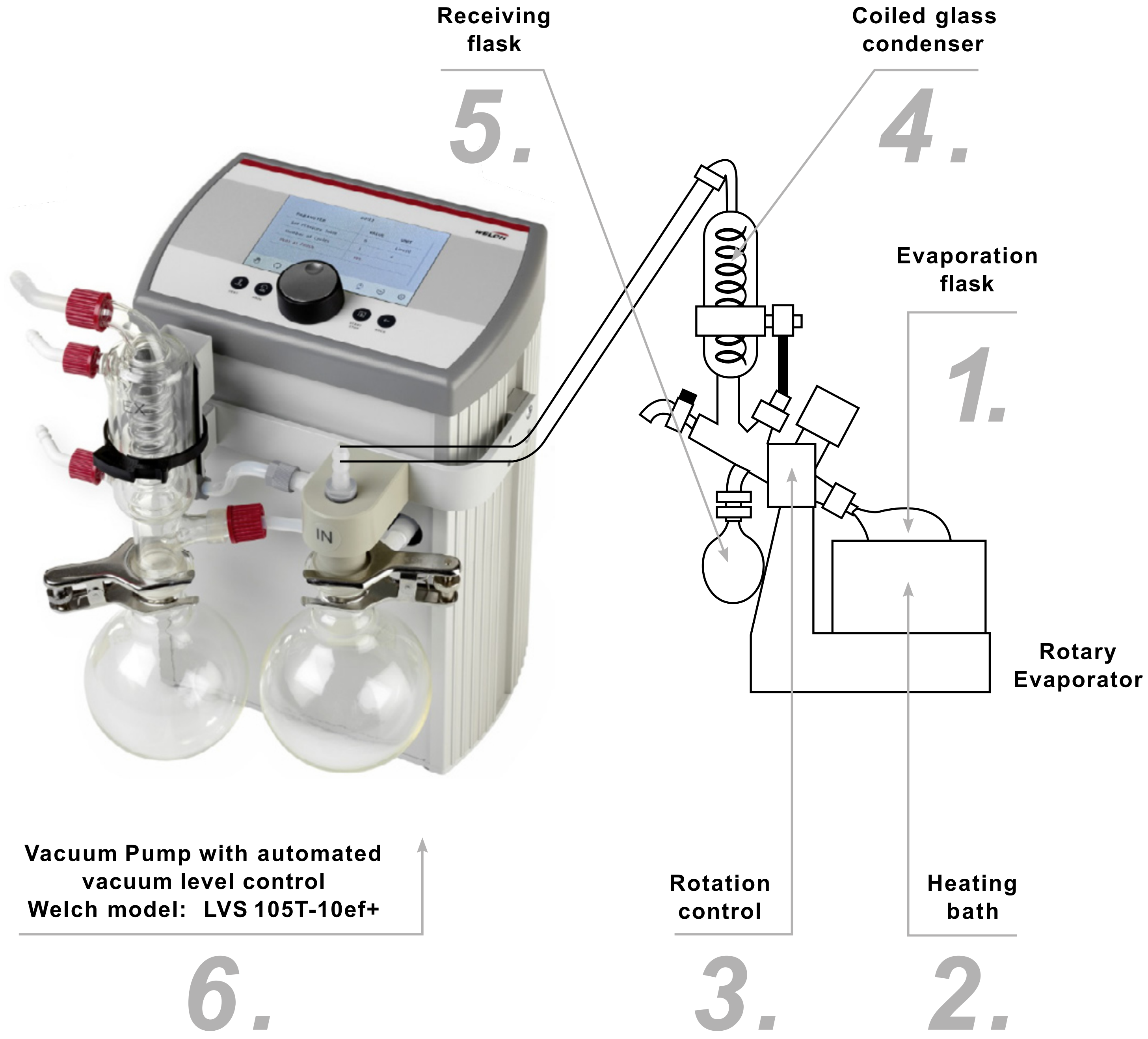
The working principle of a rotary evaporator is based on the fact that decreasing the pressure above a liquid mixture sample results in the lowering of the boiling points of the mixture’s components. The lowering of the boiling point allows quick, but gentle removal of solvents from a sample. Lowering the boiling point of a liquid mixture is particularly important for heat sensitive samples.

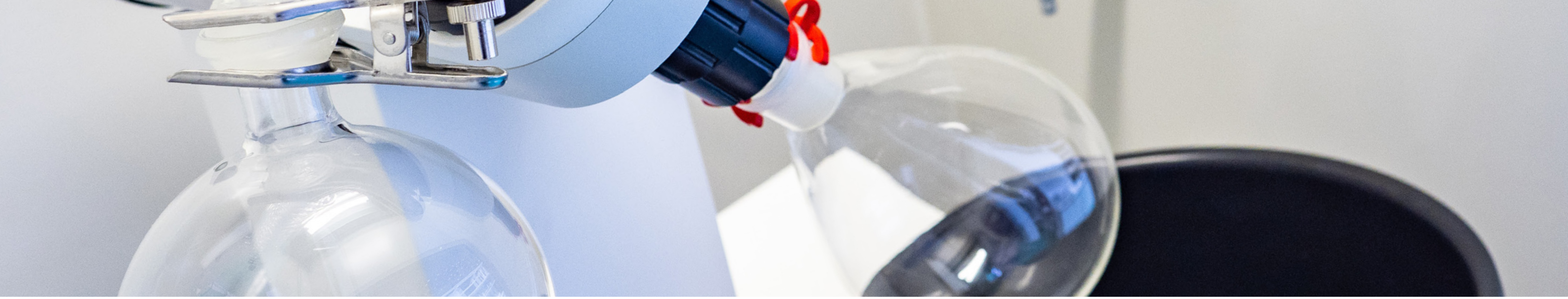
The liquid mixture is placed in an **evaporation flask (1)** warmed with **heated bath (2)**, with the **rotation (3)** ensuring that the liquid surface area is increased and evaporation proceeds more rapidly. As vacuum leads solvents to boil at lower temperatures than when they are exposed to normal atmospheric pressure, evaporating high boiling point solvents is possible without setting the bath to a high temperature that may lead to the sample’s thermal degradation.

The solvent vapors move through a vacuum-tight duct towards a **condenser (4)** where the vapor is condensed and recovered in a **receiving flask (5).** Condensers are available in various types depending on the application. Two common types found in bench scale processes are coiled glass condensers and cold finger glass condensers. The cold finger glass condensers use a refrigerant such as dry ice/isopropyl slurry.

A vacuumpump (6) is used to reduce the pressure in the rotary evaporator. The most commonly used type of vacuum pump is a chemical duty diaphragm pump. Chemical duty diaphragm pumps are used since some solvent vapors pass through the condenser into the vacuum pump and out its exhaust. The lowest pressure achievable in a rotary evaporator is typically around 1-2 Torr (1.3to 2.7 mbar) since there is always some slight leakage of room air into the rotary evaporator. The leakage is due to leaks at the rotary seal and other joints in the set-up.

If the vacuum pressure in the rotary evaporator is too low for a given solution, it can lead to bumping and foaming of the sample. The bumping and foaming is caused by pressure going well below the boiling point of sample at the set bath temperature. Vacuum regulation is critical to minimizing this unwelcome effect that will lead to sample loss. The regulation of vacuum is done manually or is automated.





KEEPING THE BALANCE

Successof the entire process is measured by the amount and quality of the desired product obtained through evaporation, as well as by the length of time required to complete the procedure. All of these successfactors are negatively affected when the evaporation and condensation processes are taking place too slowly or too fast, and are not properly balanced against each other. Key factors in finding the proper balance include bath temperature, condenser temperature, vacuum level and rotational speed of the evaporation flask.

While it may be tempting to try to achievemaximum throughput by applying higher temperatures and deeper vacuum to maximize the evaporation rate, such an approach actually introduces a number of serious process risks. For example, excessive temperature and vacuum levels may damage the compound of interest by leading samples to bump and foam, and thus drastically lower the amount and quality of the end product. They can also translate into a higher solvent loss rate.

When the evaporation rate is higher than the condensation rate, the imbalance creates the need for an oversized pump to handle the excessive solvent vapor and results in a larger portion of the solvent not being recovered. Both of these unwelcome results make the entire process more expensive. The best way to ensure a well-balanced, controlled rotary evaporation process is to select a high-quality rotary evaporator with a properly sized pump and a vacuum control solution that will help maintain the optimal vacuum level and prevent any process inefficiencies.

The rule of thumb commonly used to set the condenser and bath temperatures in a rotary evaporator is the 20/40/60 rule (also called the 20 °C temperature delta rule). For example, the condenser can be set at 20 °C, bath temperature set at 60 °C and the vacuum is adjusted so that the solvent vapor temperature is 40 °C. For low boiling point liquid mixtures, this rule of thumb will prevent foaming and bumping and will ensure reasonable solvent removal rates.

Due to heat sensitivity of some samples, it can become necessaryfor bath temperatures to be 40 °C or lower. As a result, the rule of thumb may not be followed when using a glass coiled condenser. Low bath temperatures may lead to very slow distillation rates (i.e. milliliters of solvent removed per minute) for high boiling point solvents such as DMF unless the rotary evaporator is run near its lowest achievable vacuum pressure (1-2 Torr or 1.3to 2.7 mbar).

AUTOMATING THE FLOW TO VARY VACUUM LEVEL

With vacuum pumps being the most critical components of rotary evaporators, reliance on an experienced vacuum industry partner is the key to ensuring the right equipment for your rotary evaporation procedures.

Welch, a brand of Ingersoll Rand, the world's leading provider of pressure and vacuum solutions for numerous industries, offers state-of-the-art laboratory vacuum systems (LVS) for solvent evaporation applications that include an oil-free, chemical duty diaphragm pump, a vapor condenser and optional control and liquid containment features. Our portfolio comprises a wide range of vacuum pumps and systems that are suitable for different flask sizes, vacuum levels and flow rates, and which thus meet the various needs of all kinds of rotary evaporation processes. They include the new LVS 105T-10ef+ chemistry vacuum system that with its ultimate vacuum pressure of 1.5Torr (=<2 mbar) and maximum pumping speed of 18 l/min provides an industry-leading vacuum solution for the stripping of the DMF solvent in benchtop rotary evaporator applications.

The LVS 105T-10ef+, which is already in use at major pharmaceutical companies and university laboratories around the world, includes an MPC series chemical-resistant diaphragm pump with BLDC EC motor technology, the new VCpro 600 series controller featuring rpm motor speed control (Ecoflex technology) with manual, automatic and fully programmable modes for novel processes, a liquid separator and an exhaust emissions condenser. It is ideal for the evaporation of both high and low boiling point solvents since the pump's flow is adjusted to reach the best operating vacuum pressure for the distillation.

The system stands out through its compact and portable design (WxDxH: 250mm / 260mm/ 435mm) and low power consumption. The pump provides deep stable vacuum with its flow speed control helping to adjust the vacuum level. An optimized vacuum level ensures accurate and smooth evaporation without sample bumping and foaming. The automatic function adjusts the pumping speed to match vapor load, which along with the built-in solvent recovery function allows for a fast and efficient process with high-quality distillation and solvent concentration.

ACHIEVING FAST STRIPPING OF DMF AT LOW BATH TEMPERATURES

A common misconception is that vacuum pumps with higher maximum pumping speeds will cause the evaporation rate to be higher in bench scale rotary evaporators. A comparison between the LVS 105Tef+ (max flow 18 lpm, 1.5 Torr (=<2 mbar) and a competitive pump (max flow 28 lpm, 1.5 Torr (=<2 mbar) and a competitive pump showed no difference in evaporation rate when operating under identical rotary evaporator settings. The reason for a lack of difference in DMF evaporating rate between the two pumps is that the volume of a bench scale rotary evaporator is only 4 liters (including the evaporating flask, the condenser and the receiving flask) and the solvent vapors are condensed and collected in the receiving flask, rather than pass through the vacuum pump.

When a user needs to operate at a low bath temperature, the LVS 105T-10ef+ vacuum pump is the perfect partner for a bench scale rotary evaporator. The reason is that the pump offers both deep vacuum capability (1.5 Torr/2 mbar) and automated vacuum control. Combined with an optimized rotation speed of the evaporation flask, short process times are achievable. In a test carried out under operating conditions including a 1-liter evaporating flask with a 100-ml sample, bath temperature at 37 °C, condenser temperature amounting to 10 °C, the DMF was evaporated at 3.1 ml/min at 250 rpm. For comparison, at rotational speed of 100 rpm the evaporation rate was 2 ml per minute for DMF.

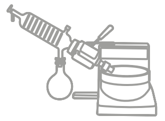
By using the innovative hysteresis-free Ecoflex vacuum control technology, Welch's LVS 105T-10ef+ chemical vacuum system ensures fully automated and perfectly balanced solvent distillation and concentration processes, delivering high-quality yield and preventing unwelcome sample-damaging effects. The system's fast, accurate and quiet operation, combined with the energy efficiency guaranteed by its BLDC EC drive, makes it the ideal partner for your benchtop rotary evaporator application.

THE PERFECT PARTNER FOR YOUR ROTOVAP

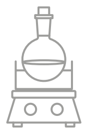
- Fast process times – high control accuracy
- Bench space saving design & Whisper quiet operation
- Multilingual high contrast 5" colour display with Light / Dark mode

APPLICATIONS

ROTARY EVAPORATOR



DISTILLATION



YOUR BENEFITS

- Smooth efficient distillation
- Fast process times – up to 40 % increase in evaporation rates
- Save energy with ecoflex speed control – GREEN by design

GREEN By Design

Ingersoll Rand

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Welch is a recognized provider of vacuum pumps, systems, and fittings for industrial, life sciences, and laboratory applications. Welch designs, manufactures, and delivers products meeting the strictest requirements for mission-critical applications, and provide a comprehensive offering of products and great engineering capabilities to fit our customers' needs. As a reliable and trusted partner for OEMs, Welch stand up to the challenge of supplying best-in-class products and solutions on time to manufacturers of equipment in the life sciences, healthcare, and industrial segments.

Please check out all our brands for your mission-critical flow control technologies:

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