Application Note
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# Accurate Temperature Control for the Separation of Solvents from Liquid Samples

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**Key Words:**

- Evaporator
- Condenser
- Solvents
- Liquid separation
- Rotary
- Rotary evaporator
- Bath circulator
- Evaporation

## Abstract

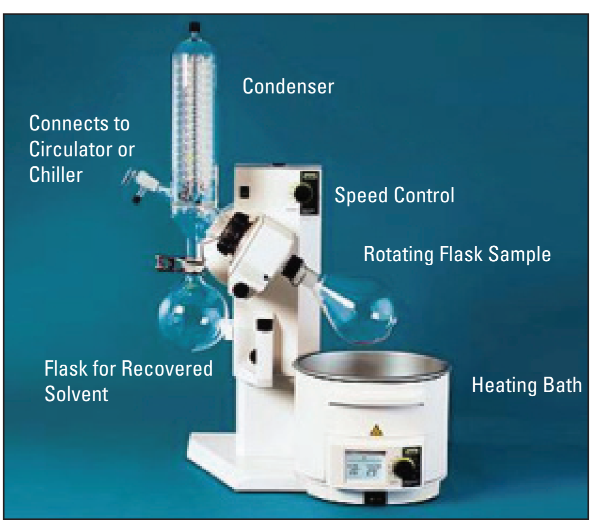
As a widely used technique, the separation of one component from a liquid sample is integral to the medicinal chemistry, pharmaceutical, chromatography and petrochemical fields. However, achieving such separations in a fast and accurate manner can prove problematic. Traditionally, this process has been time consuming and requires evaporator apparatus, e.g., rotary evaporators along with highly precise temperature control to ensure that the separated solvent is in fact the component of interest. A wide range of heated and cooled Thermo Scientific water baths and chillers are available, including the new Thermo Scientific refrigerated and heated bath circulators and the Thermo Scientific ThermoFlex series of re-circulating chillers, which can be used to effectively maintain the precise temperatures during exothermic and endothermic stages of compound separation.

## Introduction

Temperature control is an important issue across many different research processes, from chromatography and drug development to petrochemical and materials manufacturing. This is because the speed and accuracy of many chemical reactions, including changes of state, are affected by temperature alterations and often have an optimal temperature. By using a water source that is not temperature controlled, conditions are not accurately optimized to maximize experimental efficiency. As a result, methods of temperature control are extremely important in ensuring data precision. One such technique in which temperature control is extremely important is the separation of a specific solvent from a liquid sample.

In both chromatography and countercurrent distribution, the rapid quantitative recovery of a solute from a large liquid volume can be problematic. Chemical analysis studies often rely on the separation of solvents for the identification of various components of both natural and artificial materials. Such identification can lead to: the detection of:
(1) contaminants in wastewater
(2) radiopharmaceuticals for therapy and diagnosis
(3) mutagenesis in areas of petrochemical contamination
(4) identification and/or refinement of potential pharmaceuticals.

In order to facilitate the streamlining and increase the throughput of these processes, the rotary evaporator was developed.




Typical Rotary Evaporator

### The rotary evaporator

Designed for rapid separation, the rotary evaporator consists of a number of specialized components. A refrigerated water bath with a heating element is employed to ensure that a consistent temperature is maintained. If non-temperature controlled tap or facility water is used, variations throughout the day, month or year could result in a decrease in yields and/or purity of the separated solvent. The liquid sample is held in a flask, which is partially submerged in the water bath, and powered by a motor which slowly rotates the flask for an even heat distribution. This heat energy is effectively converted to kinetic energy to alter the phase of the desired component, resulting in its evaporation. In addition, the centrifugal force established from this steady rotation causes a film of warm solvent to form over the large surface area inside the flask. The solvent is therefore optimally placed to benefit from the heating effects of the water bath, improving the efficiency of the evaporation process. A vapor duct acts as both the axis of sample rotation and as a vacuum-tight conduit for the vapor being drawn off the sample. Connected to this, the vacuum pump substantially reduces pressure within the system and consequently decreases the boiling point of the solvent, to increase the speed of the reaction. Once evaporated, the solvent consequently needs to be condensed to its liquid form and collected. The temperature of the condenser apparatus is often regulated by a refrigerated circulator. The condenser itself consists of either a coil, through which the coolant passes, or a cold finger, into which dry ice or acetone, for example, are placed. Within this system, temperature control is pivotal in ensuring that the water bath is maintained at the correct heat level so as only the desired solvent is evaporated and passed to the condenser, which must remain cool enough for all of the solvent to be condensed without any loss of sample.

When temperature and pressure are properly adjusted, there is little or no tendency for unpredicted boiling (bumping) to occur (1). The forces created by the flask rotation suppress the occurrence of violent bumping, which can result in the loss of a portion of the material intended to be retained for further analysis. Additional measures can also be implemented to reduce this risk, including taking homogenous phases into evaporation, or carefully regulating the strength of the vacuum and bath temperature to ensure steady rates of phase change.

As an endothermic reaction, changing phase from a liquid to a gas consumes heat energy and therefore requires a constant heat source to maintain the specified temperature throughout the reaction. Conversely, the phase change from gas to liquid is an exothermic reaction and emits more heat energy than it consumes. A cooling source is therefore required to ensure that the excess heat energy is dissipated without affecting the reaction temperature. Maintaining these two different temperature levels during rotary evaporation is vital, however a simple equation can be used to ensure that the correct equipment is selected to accommodate the requirements of each different application.

## Methods

When selecting temperature control, it is advised that researchers adhere to the 20° rule: the condenser should be set 20 °C lower and the bath should be set 20° higher than the vapor pressure temperature (the temperature at which an equilibrium is reached between the liquid state and vapor state). An appropriately sized refrigerated circulator should consequently be selected to match or exceed the heating element wattage of the rotary evaporator bath at the specified condenser temperature.

In order to accurately maintain temperature and make efficient use of the supplied heat energy, the specific latent heat of vaporization needs to be calculated. This value refers to the amount of heat energy required to convert 1 kg of a substance from a liquid to a gas, or conversely the amount of heat energy to be removed for a gas to change to the liquid phase (the latent heat of condensation). This phase conversion requires more energy than it does to simply change temperature.

If the volume of condensate collected over a given time period is known, along with the required condenser temperature and the latent heat of condensation, then the heat load can be easily calculated:

If 0.25 liters of ethanol is collected in 0.5 hours, then 846 kJ/kg (364 Btu/lb) are required to vaporize the ethanol at standard atmospheric pressure.

- In order to calculate the heat load - the amount of energy released (as a result of an exothermic reaction) or absorbed (as a result of an endothermic reaction) - during the phase change, the following formula can be used:
- **Q = m x L**
- *Q is the heat load, m is the mass of the substance (in kg or lb) and L is the specific latent heat*

## Results

Researchers using temperature control generally work in watts, therefore any values measured in kJ are multiplied by 0.28 to convert them to watts. The density of ethanol is 0.789 kg/liter, therefore:

- Q = 0.789 kg/liter x 0.25 liter x 846 kJ/kg
- Q = 166.87 kJ x 0.28 watts/kJ
- Q = 46.73 watts

Since watts refer to watts-hour, the result needs to be corrected for time (t).

- Q/t = 46.73 watts-hour/0.5 hours
- Q/t = 93.46 watts

In this type of reaction, a relatively small heat load is generated, which is easily dissipated by a refrigerated re-circulating bath, such as the Arctic Series of refrigerated bath circulators from Thermo Scientific. In many cases it may be possible to connect multiple rotary evaporators to one bath circulator. However, with uses across such a wide variety of laboratories, rotary evaporator systems can be very large, with the capability to evaporate and condense many liters per hour, which will require the use of a much larger chiller, such as the ThermoFlex series.

## Conclusion

Since many reactions are temperature sensitive, it is vital that the correct temperature control equipment is used to fit the needs of each application. If the water is not temperature controlled, then not all parameters would be optimized. This could potentially decrease the volume of solvent obtained as well as the purity of the solvent. As a temperature dependent system, rotary evaporators effectively separate one or more specific solvents from suspension, using a combination of evaporation and condensation. In order to be sure that the separated solvent is the component of interest, the evaporation temperature must be accurately maintained, making temperature controlled water an integral component. Since this is an endothermic reaction, the water bath temperature needs to be monitored and maintained at a constant level. During the condensation phase, the reaction is exothermic and this generated heat (the latent heat of condensation) can hinder experimental accuracy. As such, calculating the heat load for each reaction will provide the user with enough information to select the most appropriately sized re-circulating chiller to maintain an optimal environment. The Thermo Scientific portfolio of water baths, re-circulating chillers and advanced thermostats is available to accurately monitor and maintain reaction temperatures.

- Non-temperature controlled water, i.e., tap water, cannot effectively maintain optimal evaporation/condensation conditions and will therefore hinder experimental integrity
- Using temperature control equipment can reduce costs and limit the environmental impact of using tap water to drain. *When using tap water, water usage and sewage fees can often justify the payback for purchasing temperature control equipment.*
- Evaporation and condensation temperatures (the water bath and re-circulating chiller temperatures) must be precisely maintained to ensure accuracy
- Calculating the heat load will enable researchers to select the most appropriate temperature control equipment

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