Vacuum Pump Operation Guide

- Plug pumps directly into outlets. **Do not** plug into extension cords or power strips.
- Store pumps in vacuum pump cabinets (if available) or other ventilated cabinet.
- Frequently check that cords and plugs are free of defects.
- Always keep belt guards in place.
- Place pumps on a tray or in secondary containment in case of oil spills.
- Shield any glassware when under vacuum.
- Only use heavy-walled round-bottom glassware for vacuum operations except for other glassware specifically designed for vacuum (ex. Erlenmeyer filtration flasks).
- Inspect glassware before and after vacuum and discard any that is scratched, chipped, broken or otherwise stressed.
- Used pump oil may be contaminated and should be disposed of as hazardous waste.
- Make sure pump oil is compatible with the vapors that will pass through the pump (i.e. do not use hydrocarbon pump oil with oxidizing gases or vapors).
- Keep combustibles away from pumps—diffusion pumps contain oil at very high temps.
- Use a **cold trap** (see below) to prevent contamination of pump oil

  Cold traps can help minimize the amount of volatile chemicals reaching the pump oil. Pump oil breaks down when exposed to high concentrations of solvents in vacuum line resulting in pump damage. Cold traps should be placed between the pump and the experiment. It is recommended two have a second cold trap for added protection. Cold traps should be large enough and cold enough to condense experimental vapors. Traps should be maintained during experiment, frequently checked for blockage, and emptied immediately following evaporation to eliminate the risk of solvents evaporating as condenser warms to room temp.
- Make sure pump inlet and outlet connections are properly attached—reversing the flow direction can build pressure within and lead to rupture, failure of vessel, or oil contamination.
- Pumps connected to **rotovaps** (see below) should be located in a fume hood, vent to specified lab exhaust, or be equipped with sufficient condensers and traps to prevent escape of solvent vapors into lab space.

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Vacuum pumps can serve several purposes in a chemical laboratory but are commonly used with rotary evaporators. The following are quick guides provided by Buchi to ensure users are operating rotovaps properly and selecting appropriate temperature and pressure for a given solvent.

Your Evaporation Guide
Operation – Temperature difference

Achieve higher distillation efficiency when using a rotary evaporator – Impact of temperature differences

Summary

There is a direct relationship between the heating bath temperature and the evaporation rate. The more energy applied to the evaporation side, and at the same time removed from the condensation side, the more efficient is the distillation. Furthermore, sufficient cooling as well as an appropriate and stable under pressure are crucial for efficient distillation. On the other hand, the consumption of electrical energy is comparatively greater at higher temperatures. Moreover, some samples are thermo-sensitive, thus exacerbating the circumstances. Therefore the respective parameters have to be fine-tuned to the individual sample and application. The "Delta 20 Rule" is a guideline to compromise between high evaporation output and energy usage. For instance, using the 10/30/50 parameters is appropriate for the evaporation process in order to bring in and to carry off the accumulated energy efficiently.

Introduction

The performance of a rotary evaporator is limited by the input, the amount of heat that can be added to the evaporation side, and the output, the amount of heat that can be removed on the condensation side. Basically, energy is imparted to the solvent in order to transform it to the vaporous state; during the condensation cycle this energy has to be removed again within the same length of time.

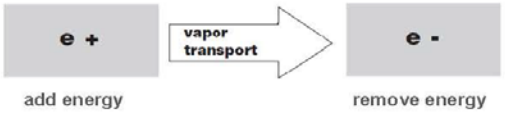

Figure 1: *Schematic representation of the evaporation-condensation process. Heating → evaporation; cooling → condensation.*

Formerly, only the energy supply was easily controllable. The cooling temperature was rather inflexible as mainly tap water was used as the cooling source. Moreover, the vacuum was only roughly controllable.

Nowadays, the vacuum can be adjusted very precisely and kept stable. Furthermore, with the possibility of the modern "recirculating chiller", the energy supplied for cooling the condenser can be selected accurately, typically to produce temperatures as low as -5 to 10 °C. Therefore recirculating chillers are very effectively in cooling and the distillation can be kept at low temperatures.

The heating bath temperature, the vacuum as well as the cooling temperature need to be adjusted to the condenser's capacity. A condenser is working at its optimum capacity if two-thirds of its height is covered with condensate, hence the top third acts as a safety barrier for "entrained" low-boiling solvent plus for pressure fluctuations. A condenser is overloaded if condensate is seen to form downstream from the condenser or if the vacuum pump sucks continually in order to maintain a specific pressure. The speed of evaporation and condensation should be attuned to maintain a balanced dynamic pressure.

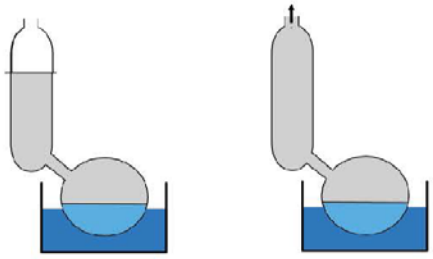

Figure 2: *Illustration of the optimal utilization of condenser's capacity (left); condenser is overloaded → loss of solvent (right).*

When working with a thermo-sensitive sample, a mild heating bath temperature needs to be selected in order not to harm the compounds. In addition, a heating bath at lower temperature is more convenient to work with. For instance, with a heating bath temperature of 50 °C, the evaporating flask can be changed without any risk of scalding. With higher temperatures, the vaporizing rate of the heating bath medium (e.g. water) increases, and it thus has to be refilled more frequently. This results in additional consumption of energy.

Experiment

The aspects of heating and cooling are very important and determine the evaporation rate.

It is interesting to examine to what extend different heating bath temperatures influence the evaporation output. The aim of the following experiment was to analyze the impact of the amount of energy, in form of heat, applied to the system on evaporation rate of a solvent single-stage distillation. The experiment was executed with a BUCHI Rotavapor®.

For the experiment the evaporation process was executed using five different heating bath temperatures (from 40 to 80 °C).

Parameter

Solvent	acetone
Pressure	556 mbar
Vapor temperature	30 °C
Cooling temperature	7 °C
Flask size	1 L
Content	500 mL
Immersion depth	fill level

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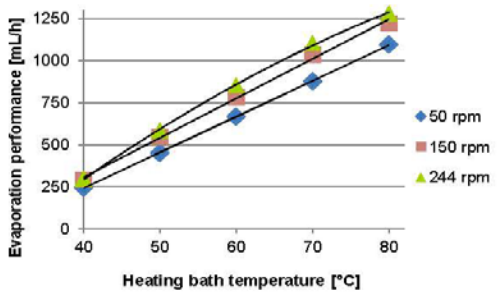
Result



Graphic 1: Illustration of the influence of the bath temperature on the evaporation output.

As illustrated in the above graphic, the higher the heating bath temperature, the higher is the evaporation rate. The differences of the evaporation output increased more or less linearly with the temperature rise. For instance, with a heating bath of 80 °C, the distillation output was about four times greater compared to a heating bath temperature of 40 °C.

Interpretation

As the temperature of the heating bath was raised, the evaporation output increased significantly. However, the energy consumption of the heating bath and recirculating chiller increased, too. For instance, when using an 80 °C heating bath, it should be remembered that much more energy has to be supplied and again removed from the system than is the case when working at lower temperatures.

Recommendation

The heating bath temperature and the vacuum needs to be coordinated for the condenser to work as closely as possible to optimum condenser's capacity without being overloaded. For a sufficient condensation of the vapor, the cooling temperature should be about 20 °C lower than the vapor temperature.

BUCHI recommends that the "Delta 20 Rule" should be applied. This rule of thumb can be applied as following: set the bath temperature at 50 °C to yield a solvent vapor temperature of 30 °C, which is subsequently condensed at 10 °C [1].

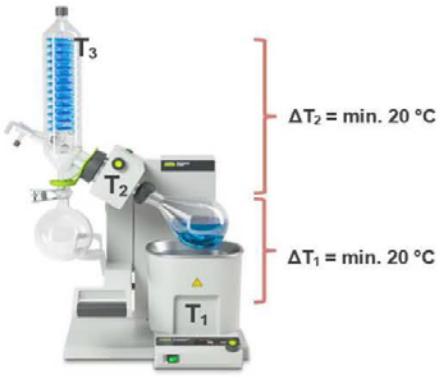


Figure 1: Representation of the "Delta 20 Rule", the parameters 10/30/50 applied.

The "Delta 20 Rule" can also be applied to lower heating bath temperatures for solvents with a low boiling point or thermo-sensitive products. For example: cooling media: 0 °C; vapor: 20 °C; heating bath: 40 °C and the pressure lowered in order to lower the solvents boiling temperature.

A heating bath temperature above 50 °C is less easy to handle, thus increasing the risk of accidents. Moreover, environmental and economical issues should also be taken into account. The "Delta 20 Rule" makes solvent removal simple and efficient. The vacuum is the only setting that has to be changed and the pressure for each solvent can be conveniently selected from BUCHI's "List of solvents".

The Heating Bath B-100 and B-305 have a heating power of 1300 watts and a standard condenser a cooling area of 1500 cm^2 [2], hence it can achieve high evaporation output.

To sum up, the "Delta 20 Rule" compromises evaporation output and energy consumption. The optimized settings of the heating and cooling temperatures are depending on the specific application and have to be fine-tuned for each individual sample.

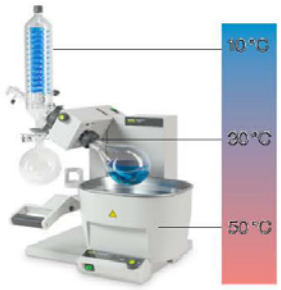
References

[1] BUCHI - Training Paper, Distillation with a Rotary Evaporator
[2] Technical Datasheet, Rotavapor® R-300

Increase your distillation efficiency

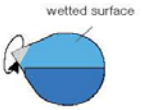
The following tips help you improve the efficiency of your evaporation process, to save time, to conserve energy and to reduce the environmental impact.

Δ 20 °C rule - 10/30/50 °C



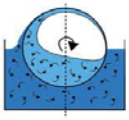
1. Set heating bath temperature 50 °C
2. Cooling water temperature 10 °C or lower
3. Adjust needed vacuum for a boiling point of 30 °C according to the list of solvents

Immersion angle



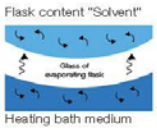
Use standard position (25 °) for best efficiency without jeopardizing the sample

Rotation speed



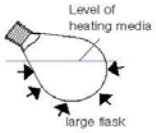
Use 250 to 280 rpm for maximum turbulence at high durability

Flask thickness



Use 1.8 mm thick flasks (1 L) for best temperature exchange at high safety

Flask sizes



Select a flask that accommodates approximately twice the starting sample volume

Download the comprehensive evaporation guide white papers:
www.buchi.com/application

List of Solvents
Evaporation

Solvent	Formula	Vacuum*
Acetic acid	$C_2H_4O_2$	26
Acetone	C_3H_6O	370
Acetonitrile	C_2H_3N	153
Benzene	C_6H_6	162
n-Amylalcohol, *n*-pentanol	$C_5H_{12}O$	6
n-Butanol	$C_4H_{10}O$	14
tert-Butanol, 2-methyl-2-propanol	$C_4H_{10}O$	78
Chlorobenzene	C_6H_5Cl	22
Chloroform	$CHCl_3$	332
Cyclohexane	C_6H_{12}	154
Dichloromethane, methylene chloride	CH_2Cl_2	699
Diethylether	$C_4H_{10}O$	838
trans-1,2-Dichloroethylene	$C_2H_2Cl_2$	317
Diisopropylether	$C_6H_{14}O$	251
Dioxane	$C_4H_8O_2$	68
Dimethylformamide (DMF)	C_3H_7NO	6
Ethanol	C_2H_6O	97
Ethylacetate	$C_4H_8O_2$	153
Heptane	C_7H_{16}	77
Hexane	C_6H_{14}	264
Isopropyl alcohol	C_3H_8O	78
Isoamyl alcohol	$C_5H_{12}O$	9
Methanol	CH_4O	218
Pentane	C_5H_{12}	834
Propionic acid	C_3H_6O	8
n-Propylalcohol	C_3H_8O	37
Pentachloroethane	C_2HCl_5	8
1,1, 2,2-Tetrachloroethane	$C_2H_2Cl_4$	16
1,1,1-Trichloroethane	$C_2H_3Cl_3$	204
Tetrachloromethane	CCl_4	179
Tetrahydrofurane (THF)	C_4H_8O	249
Toluene	C_7H_8	48
Trichloroethylene	C_2HCl_3	119
Water	H_2O	42
Xylene	C_8H_{10}	15

*Pressure in mbar for boiling point at 30 °C (heating bath 50 °C)

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