Training Papers
Distillation with a Rotary Evaporator

Contents

1	**Introduction**
2	**What is a rotary evaporator?**
3	**Selecting the right rotary evaporator**
3.1	**Rotavapor® models**
3.2.1	**Heating bath**
3.2.2	**Heat transfer medium**
3.3.1	**Glass assemblies**
3.3.2	**Rotary evaporator RE or EL?**
3.4	**Special accessories**
4	**Working with the rotary evaporator**
4.1	**Heating/cooling**
4.2	**Pressure control**
4.3	**Evaporating flask and rotation**
5	**The thermal pump**
6	**How to distill?**
7	**Definitions**

60 YEARS BÜCHI **Progress built on tradition**

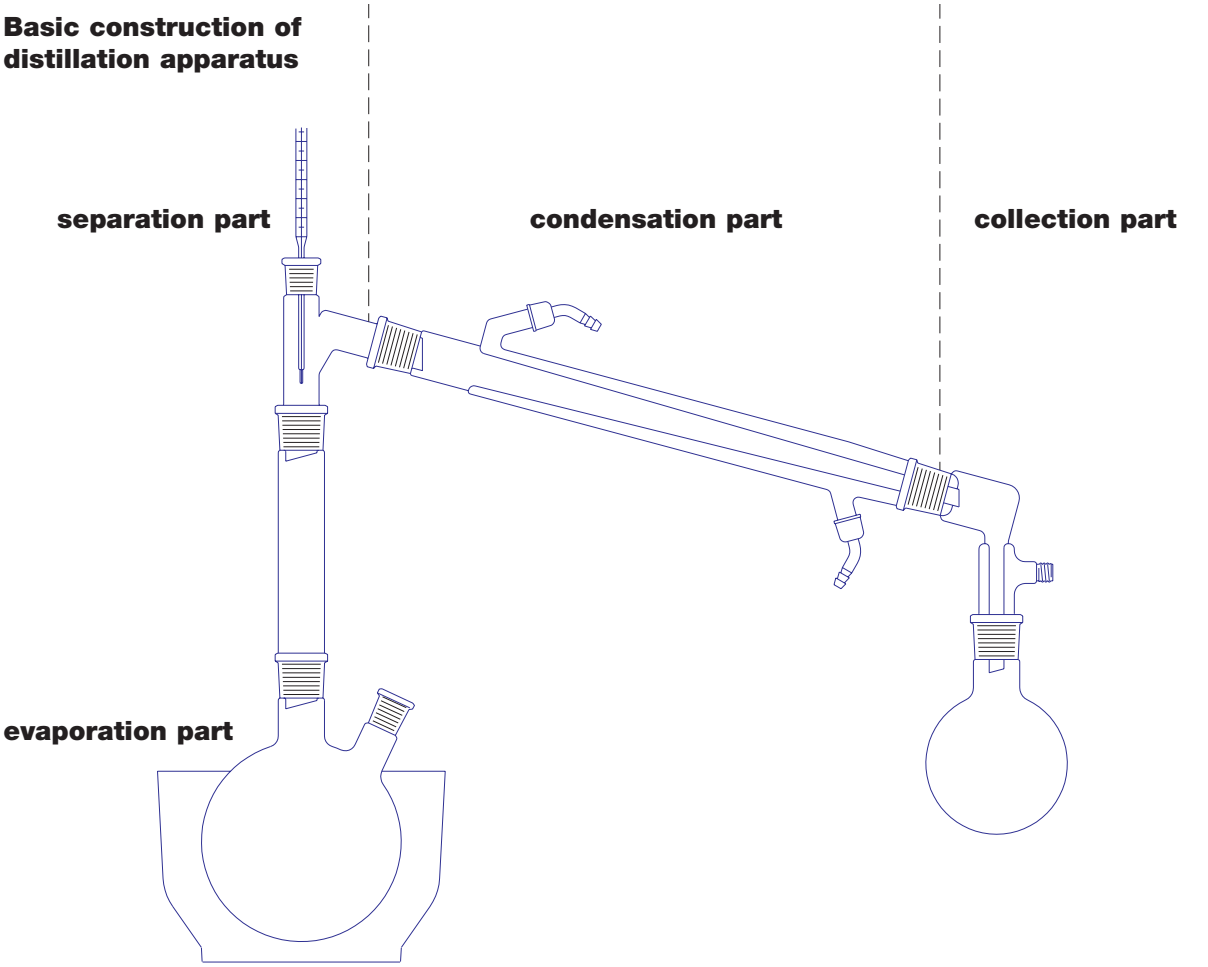
English, Version B (16 pages) **Order No.**

Distillation with a Rotary Evaporator **97743**

1 Introduction

From the beginnings of the ancient alchemists[1] to today's modern research, the principles behind simple laboratory distillation apparatus have hardly changed.

Basic construction of distillation apparatus



Evaporation part

This is where the mixture is made to evaporate by heating.

Separation/ fractional part

This is where the actual separation of solvent and non-volatile or less volatile components takes place.

Condensation/ cooling part

Originally this section was only required when the distillate was to be reused. Today, condensation should be as complete as possible for environmental reasons.

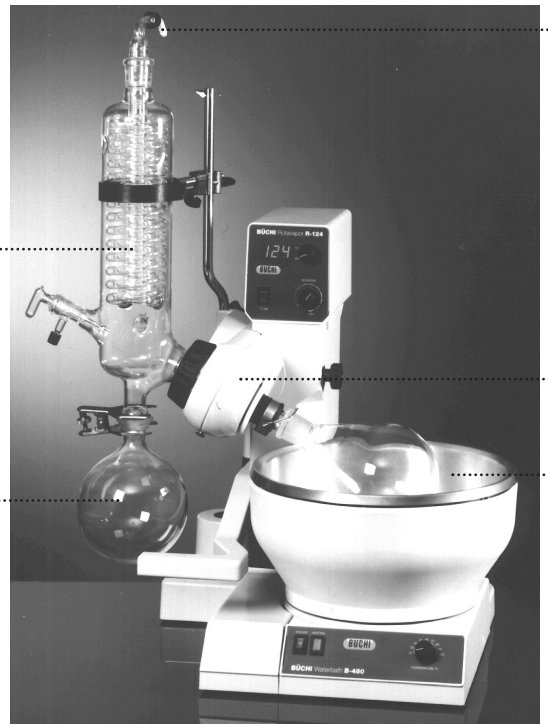
Collection part

This is where the various condensates are collected.

2 What is a rotary evaporator?

A rotary evaporator is a specially designed instrument for the evaporation of solvent (single-stage or straight distillation) under vacuum. The evaporator consists of a heating bath with a rotating flask, in which the liquid is distributed as a thin film over the hot wall surfaces and can evaporate easily. The evaporation rate is regulated by the heating bath temperature, the size of flask, the pressure of distillation and the speed of rotation.

Functional principle of a rotary evaporator



Evaporation part

The solvent is heated via the heating bath. A thin film of solvent forms on the inner surface of the rotating evaporating flask, resulting in a higher rate of evaporation. Rotation ensures homogenous mixing of the sample and prevents overheating inside the flask.

Rotary drive

The drive unit provides constant rotation of the evaporating flask for the reasons stated above.

Condensation part

The solvent vapor flows at high speed into the condensation part (condenser) of the rotary evaporator. At this point the energy inside the solvent vapor is transferred to the cooling medium (usually water) and the solvent condenses. The condensed solvent now flows by force of gravity into the receiving flask.

Receiving flask

The receiving flask is used to collect the condensed solvent.

Vacuum

Vacuum is used to lower the boiling temperature and hence raise the efficiency of the distillation process.

Advantages of the rotary evaporator (compared with static apparatus)

With a vacuum rotary evaporator you can carry out single-stage distillation runs quickly and gently.
The evaporation capacity of a rotary evaporator is about 4 times greater than that of a conventional, static distillation apparatus. Heat transmission in the heating bath as well as inside the flask is greatly improved by rotation of the evaporating flask. Rotation greatly enlarges the active surface inside the flask, hastening the evaporation. With the liquid remaining at any one point of the flask wall for a short time only, it is subject to minimum stress during the distillation (no overheating, no incrustation). Bumping and foaming are greatly reduced by the rotation.

Rotary evaporators are used for:

- Distillation of solvent
- Concentration of solutions and suspensions[2]
- Crystallization or recrystallization[3]
- Synthesis[4] and purifying of fine chemicals
- Soxhlet extraction[5]
- Powder and granules drying
- Recycling of solvent

3 Selecting the correct rotary evaporator

3.1 Rotavapor® models

3.1 Rotavapor® models

R-114

This is the most inexpensive model for daily routine work. Rotation and bath temperature can be set individually.

R-124

This model is for users who would like to have a little more information about their distillation processes. The speed is indicated on a digital display, and with an additional sensor you can also read off the bath or vapor temperature. These parameters are important for the reproducibility of results and for determining the optimum operating conditions.

R-134

This rotary evaporator has all the functions of the R-124 plus a built-in vacuum controller. Optimum conditions of distillation can be fixed by choice of the set point of pressure.
You can have the pressure, temperature or speed of the rotation displayed at any time. The vacuum controller keeps the set pressure range constant automatically. It reduces costly monitoring and operating times to a minimum and lowers the risk of foaming or bumping by ensuring stable conditions of pressure. Selection of the vacuum for each specific solvent enables practically 100% condensation in the condenser. The result is a massive reduction in the amount of solvent in the waste water.

R-144

With the same benefits as the R-134 plus a dual temperature sensor, the R-144 adds up to perfect fully automatic distillation. Pressure conditions are fixed automatically in accordance with the respective solvent. The current values of pressure, temperature and rotation can of course be called up in the display at any time during the distillation.

3.2.1 Heating bath

A heating bath is required to heat the evaporating flask. Büchi heating baths can take flask sizes from 50 ml to 3000 ml without having to be moved. Bath capacity is 5 litres. The heater is attached to the underside of the pan. The easy cleaning of the bath is therefore possible. The cool wall construction of the bath acts as protection in case of accidental contact. There are two water bath models to choose from: the B-481 with current temperature display, the B-480 without a temperature display. If demineralized or distilled water is used in the bath, 0.2% borax ($Na_2B_4O_7$ x 10 H_2O) must be added.

Front panels

B-480

B-481

B-485

3.2.2 Heat transfer medium

For certain applications it is necessary to use an oil bath. If the boiling points are below 160°C (at normal pressure), it is better for a variety of reasons to heat with a water bath.

Advantages of a water bath:

- High specific thermal capacity = high efficiency
- Good distribution of heat
- No classification of toxicity
- Readily available
- Low risk of accident

Where it is essential to use an oil bath, the choice of oil (polyethylene glycol, silicone oil, carbowax, bath oil) is very important.

Some of the criteria to be considered:

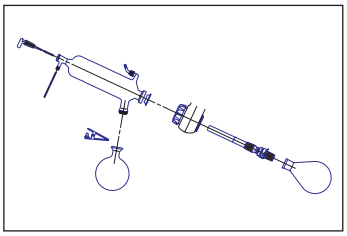
- Viscosity[6]
- Water solubility
- Polymerization[7] of the oil at higher temperatures
- Toxic vapors
- Oily precipitation on the surroundings
- Risk of accident (high temperature)
- Combustibility
- Price

Büchi recommends water-soluble polyethylene glycol (PEG).

3.3.1 Glass assemblies

A diversity of glass assemblies is available in order to obtain optimum results (throughput, separation efficiency).

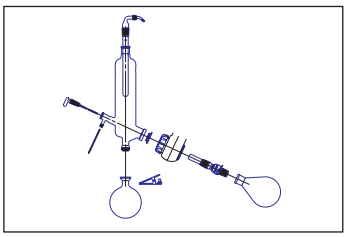
Glass assembly A (sealing system RE)



The diagonal condenser is very well suited for simple distillations at a high rate of throughput. It is the low-priced model but requires more room.

Suitable for:
- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes

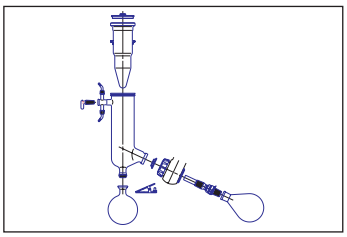
Glass assembly V (sealing system RE)



The vertical design saves space. Glass assembly V is very well suited for distillations of simple and high-boiling point solvents.

Suitable for:
- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes

Glass assembly C (sealing system RE)

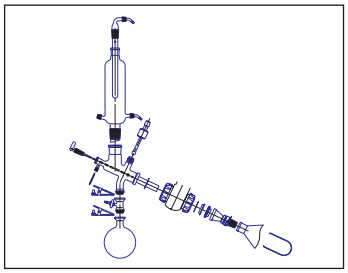


The water condenser is replaced by a cold trap that enables very low freezing temperatures. Hence, with this assembly it is also possible to completely condense such extremely volatile and low-boiling solvents as chloroform or dichloromethane. You can fill the cold trap not only with common freezing mixtures such as dry ice/acetone (-70°C) or ice/common salt (-20°C) but also with liquid nitrogen (approx. -190°C). This allows you to perform sublimations as well. The cold trap does not require running water for cooling; therefore, it saves water.

Suitable for:
- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes
- Sublimations

Glass assembly S (sealing system EL)

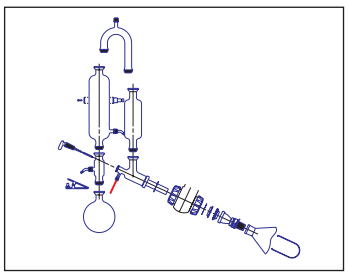
This apparatus is ideal for reflux reactions and for the distillation of high-boiling solvents such as toluene and water. Solid/liquid extractions are possible with a Soxhlet extraction unit. This arrangement is suitable for universal application.



Suitable for:
- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes
- Soxhlet extractions
- Synthesis and cleaning of fine chemicals

Glass assembly E (sealing system EL)

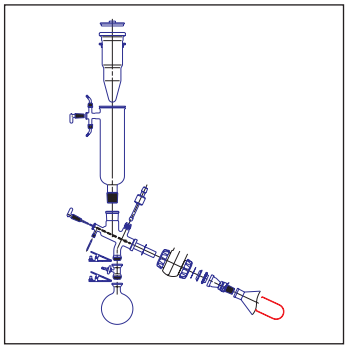
The descending condenser is particularly ideal for the distillation of solvent with low heat of vaporization, for high-foaming products and for the recovery of solvent. The expansion vessel serves as a buffer for foaming samples. A higher degree of separating efficiency is possible by adding a packing (e.g. Raschig rings[8]) in the expansion vessel.



Suitable for:
- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes
- Cleaning of fine chemicals, better separating efficiency

Glass assembly CR (sealing system EL)

With this apparatus you can also perform reflux reactions. The cold trap permits very low freezing temperatures. Hence it is also possible to completely condense such extremely volatile and low-boiling solvents as chloroform or dichloromethane. The cold trap does not require any running water ⟶ saves you water. It can be filled not only with common freezing mixtures such as dry ice/acetone (-70°C) or ice/common salt (-20°C) but also with liquid nitrogen (approx. -190°C). These low freezing temperatures allow you to perform sublimations as well.

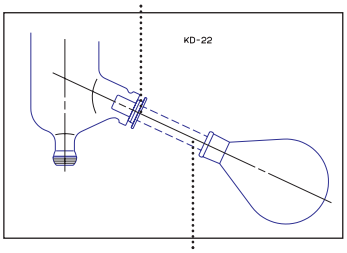


Suitable for:
- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes
- Synthesis and cleaning of fine chemicals
- Soxhlet extractions
- Sublimations

3.3.2 Rotary evaporator RE or EL?

The sealing system is determined by the glass assemblies.

KD-22 seal stationary

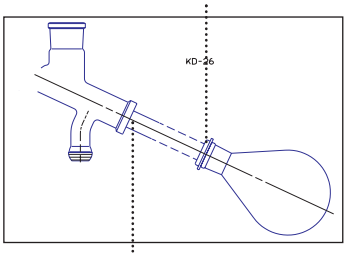


Vapor duct rotates

RE:
In the case of the RE sealing system, the seal (KD-22) is stationary and the vapor duct rotates.
Is used on glass assemblies A, V and C.

Characteristics:
- Working in reflux mode not possible
- Seal on condenser side
- Flanged evaporating flasks cannot be used

KD-26 rotates



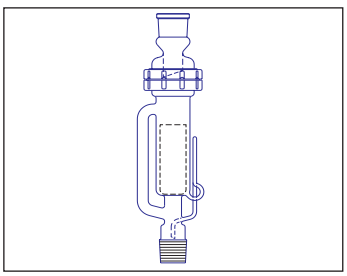
Vapor ducts stationary

EL:
In the case of the EL sealing system, the vapor duct is stationary and the seal (KD-26) rotates around the vapor duct.
Is used on glass assemblies S, E and CR.

Characteristics:
- Working in reflux mode possible with glass assemblies S and CR
- Seal is on evaporator side
- Flanged evaporating flasks can be used

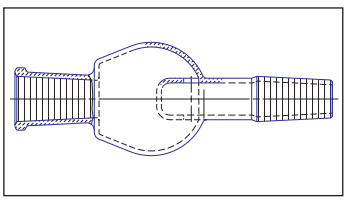
3.4 Special accessories

Soxhlet extraction unit



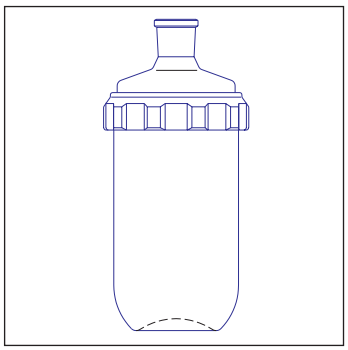
Extraction is the process of separating a substance out of a solution or a mixture of substances. The Soxhlet unit is used to perform solid/liquid extractions (e.g. peanut oil from peanuts).
The solvent is evaporated, condensed in the condenser and passes through the extraction material (in the Soxhlet unit). The dissolved product runs together with the solvent back into the evaporating flask where it is enriched. The solvent is evaporated again. At the end of the extraction process the solvent is distilled off, and the extracted substance remains in the evaporating flask. This complete process can take place in the same apparatus (glass assemblies S or CR necessary).

Reitmayer foam trap



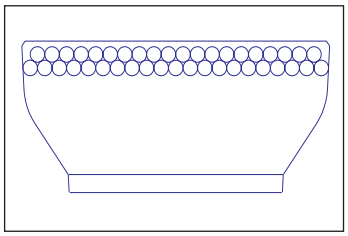
During the boiling process there is a risk of liquid particles being trapped in the rising vapor and contaminating the condensate. Intensively foaming products can have the same effect. A splash guard (Reitmayer foam trap) is used with critical substances. The foam trap is fitted between the evaporating flask and the vapor duct.

Beaker flask



The extra-wide neck of the beaker flask allows easy removal of highly viscous or solid products. Additionally, the inside of the flask can now be easily cleaned by hand. The unique large surface area and design features a cylindrical shape to enhance distillation and act as a buffer against bumping and foaming.
The beaker-like design does not require a support to stand on the bench.

Swimming balls

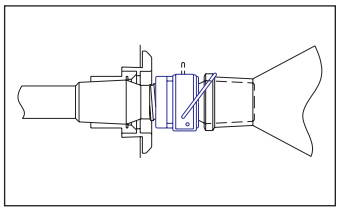


Water evaporates easily. Hot water baths can be protected from drying out by covering the surface with swimming balls.

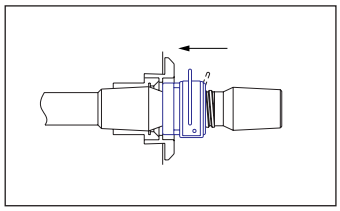
Combi-clip

The patented combi-clip performs the following **functions** in addition to holding the evaporating flask:
Turn anticlockwise to release the evaporating flask if the ground joint should jam. Turn clockwise to press out a jammed vapor duct.

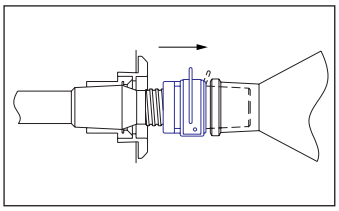
Holding the evaporating flask



Pressing out a jammed vapor duct

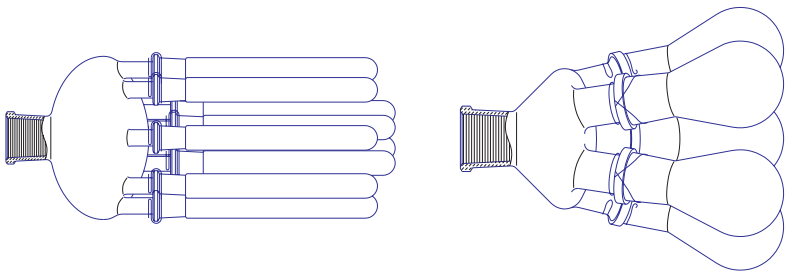


Releasing the evaporating flask if the ground joint jams



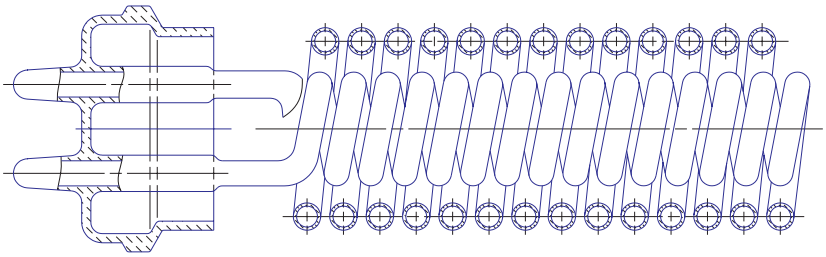
Spider evaporator

If you need to concentrate several samples under the same conditions, it makes sense to use a spider evaporator. Up to 5 samples can be concentrated simultaneously using 50 ml or 100 ml flasks. Cylindric tubes
20 ml are available for smaller volumes, permitting concentration of up to 20 samples.



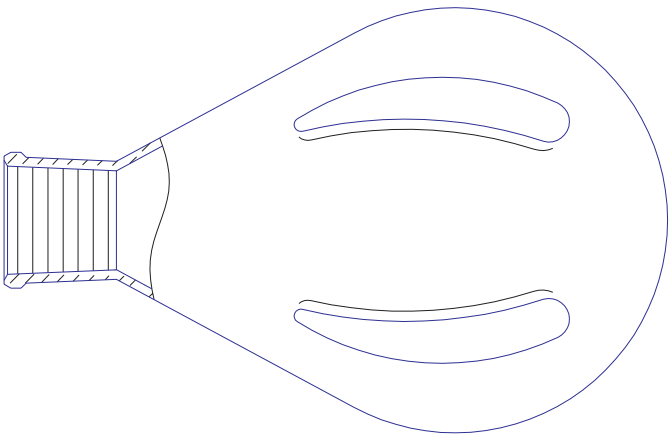
Cooling coil insert

If you want to distill higher-boiling as well as low-boiling solvents, the coil can be inserted in the cold trap in place of the cold finger. It is then possible to operate the cold trap (glass assemblies C and CR) like a normal liquid-cooled condenser.



Drying flask

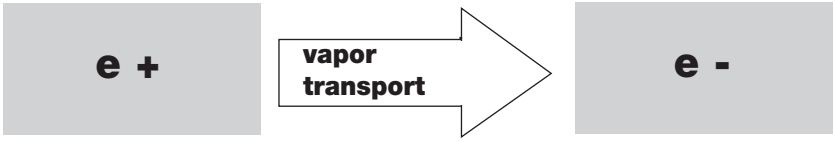
During the concentration of suspensions, the solid product is kept from caking by strong turbulence inside the drying flask. The result is a loose powder.



4 Working with the rotary evaporator

4.1 Heating/cooling

The capacity of a rotary evaporator is limited by the amount of heat that can be input on the evaporator side and the amount of heat that can be removed on the condensation side in the same period of time. A water or oil bath has a power consumption of 1300 watts. A rotary evaporator (type RE, EL) has a cooling area of 0.15m^2 (cold trap: 0.05m^2).



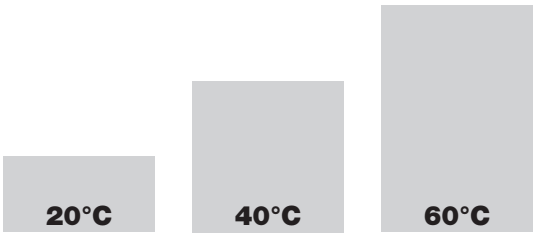
(heating/ evaporation) **(condensation/ cooling)**

For practical reasons the water bath (e.g. B-481) has established itself as first choice for the heating of laboratory rotary evaporators. Water is an environment-friendly and clean-to-use heat transfer medium. Heat distribution is good thanks to water's low viscosity, and its high specific heat capacity which means that it can absorb and pass on plenty of energy. Swimming balls can be used to reduce the exposed surface of the water as a means of lowering the rate of evaporation from the heating bath.

In general, it is not possible to choose the cooling tap water temperature. The conditions of distillation are dependent among other things on the temperature of the cooling water.

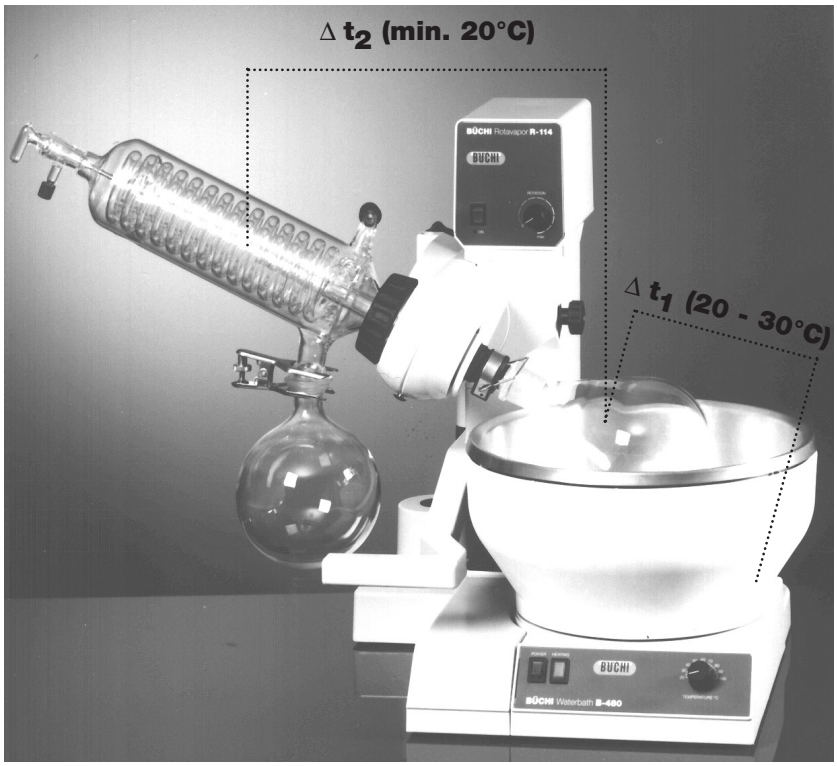
The following rule of thumb can be applied:

20°C cooling water
40°C vapor
60°C bath



Advantages of a bath temperature of 60°C

- The evaporating flask can be changed without risk of scalding.
- The rate of water evaporation from the heating bath is still not very high.
- The distillation capacity of the rotary evaporator is used at a good level of efficiency.



4.2. Pressure control

Unlike a substance's melting point, its boiling temperature depends greatly on the ambient pressure. The higher the ambient pressure, the higher the boiling temperature; the lower the ambient pressure, the lower the boiling temperature (e.g. water: 100°C in Nice at sea level, but approx 84°C on Mont Blanc at 4810 meters altitude). High-boiling substances can be distilled at a lower boiling temperature if the ambient pressure is reduced.
In practice, distillations are performed at reduced pressure (vacuum distillation) in order to prevent damage to temperature-sensitive substances. Substances with a boiling point of 100°C or higher are often distilled in vacuum in order to enable a water bath to be used as heat source (temperature range up to max. 100°C).
To apply the rule of thumb, i.e. 20 - 40 - 60°C, the pressure must be selected for the vapor temperature (and hence the boiling point) to equal 40°C. An auxiliary device called a vacuum controller (e.g. B-721) is needed to set this pressure reliably and to adjust it accordingly during the course of distillation. With the B-721 the distillation can run manual or automatically (using an automatic probe).

e.g. water:	
100°C	**1013 mbar**
80°C	**550 mbar**
60°C	**300 mbar**
40°C	**72 mbar**

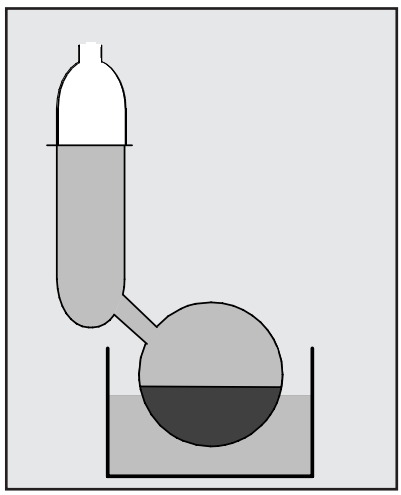
A common pump chosen to operate a rotaryevaporator is a controlled water- jet pump (e.g. B-164, B-167, B-764, B-767).
A PTFE diaphragm pump (e.g. V-500) or a vacuum system (e.g Büchi Vacuum Line V-501 to V-513) is recommended, particularly for environmental reasons (water consumption).

An apparatus with joints is never absolutely leak-proof. If you intend working with a vacuum well below normal pressure, it is advisable to subject the apparatus to a leak test in advance. This entails evacuating the apparatus and then measuring the pressure over a certain time. The leakage rate is normally quoted as the rise in pressure per unit of time.

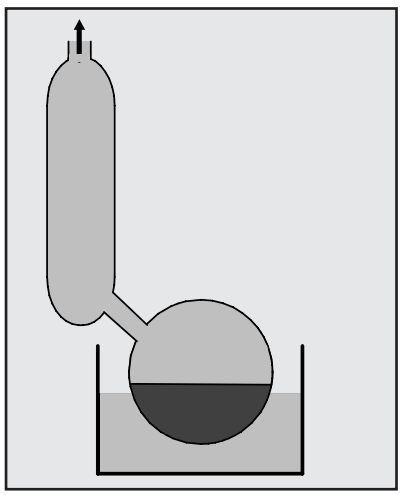
A laboratory rotary evaporator is deemed to be leak-proof when the pressure at a specified level rises by less than 10 mbar in 5 minutes.

A upper-scale rotary evaporator is deemed to be leak-proof when the pressure rises by less than 5 mbar in 15 minutes.

The condenser capacity, the vacuum and the bath temperature are coordinated for the condenser to work as closely as possible to optimum capacity without overloading. A condenser is overloaded if condensate is seen to form downstream from the condenser or if the pump sucks continually in order to maintain a specified pressure. A condenser is working to optimum capacity if two-thirds of it are covered with condensate. The last third acts as a safety barrier for pressure fluctuations on the one hand and for "entrained" low-boiling solvent (contaminant) on the other.



Optimum utilization of condenser capacity

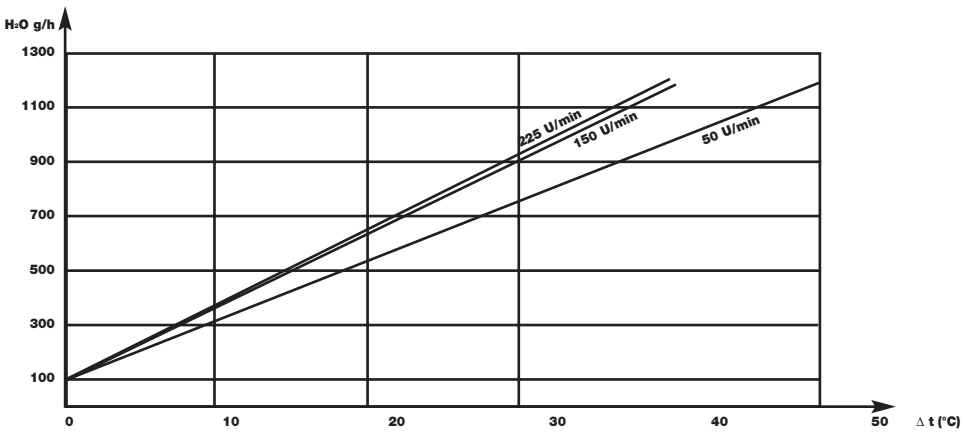


Condenser is overloaded ⟶ loss of solvent

4.3 Evaporating flask and rotation

The distinctive characteristic of a rotary evaporator is the rotating evaporating flask. Its rotation should be selected to produce maximum turbulence in the bath as well as inside the flask. This turbulence depends on the flask size, the amount of substance filled in the flask and the viscosity of the substance. As a general rule:

The higher the rate of rotation, the higher the rate of distillation



The rate of rotation can be raised to a point where the contents will be pressed against the wall by centrifugal force (as in a centrifuge) and will co-rotate with the flask. In this case the turbulence will decrease again.
⟶ **The result**: A lower rate of evaporation. A further general rule:

The bigger the evaporation flask, the higher the rate of distillation

5 The Thermal pump

At a pressure of 72 mbar (standard conditions of distillation for a rotary evaporator), 1 ml of water produces 17,500 ml of steam! In other words, a distillation apparatus continually transforms a (small) volume of liquid into a thousand to twenty-thousand times larger volume of gas in the evaporating flask. The space available for such an expansion is limited, so there is a build-up of pressure.
It is this pressure which causes the gas particles to flow from the evaporator side to the condenser side.
Assuming condensation actually takes place at the condenser, the (immense) volume of gas is retransformed abruptly into a small volume of liquid, creating a local vacuum. This difference in pressure between the evaporating flask (high pressure) and the condenser (local vacuum) causes large volumes of gases (vapors) to flow at high velocity from the evaporating flask to the condenser. This driving force is known as the **thermal pump**.
Vapor velocities at the narrowest point of rotary evaporator can exceed 150km/h.

6 How to distill?

Starting

1. Determine the bath and boiling temperature (consult tables).
2. Preheat the bath.
3. Carry out a vacuum leak test on the apparatus without solvent (see page 14).
4. Check the cooling water flow. It should be neither too high nor too low (for a laboratory rotary evaporator: 40 - 50 l/h).
5. Aspirate or fill in solvent.
6. Set the distillation pressure and evacuate.
7. Set the rotation.
8. Lower the evaporating flask into the bath. If the distillation product is inclined to foam, lower in steps.
9. Distill.

Stop

1. Stop the rotation.
2. Raise the evaporating flask out of the bath.
3. Aerate the system.
4. Switch off the heating bath.

7 Definitions

[1] Alchemists:	Chemists of the Middle Ages
[2] Suspension:	A mixture of solid substances and a liquid (e.g. soup)
[3] Recrystallization:	Method of cleaning substances able to form crystals
[4] Synthesis:	Production of complex chemical compounds from simpler compounds, e.g. hops, malt, water ⟶ beer (alcohol)
[5] Soxhlet extraction:	Removing specific components from solid mixtures of substances by dissolving the components in a solvent (e.g. tea from a tea-bag)
[6] Viscosity:	The resistance of a liquid to flow (e.g. honey)
[7] Polymerization:	Formation of longer molecules (e.g. plastic fibres)
[8] Raschig rings:	Glass accessories for increasing the efficiency of separation during distillation

BÜCHI
Labortechnik AG

P.O. Box
CH-9230 Flawil
Switzerland
Phone 071 394 63 63
Fax 071 394 65 65
http://www.buchi.com
e-mail: sales@buchi.com