

The Laboratory Assistant



The Laboratory Assistant

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«The Laboratory Assistant» – an indispensable aide for your daily laboratory work!

The stimulus for innovation is the conversion of visions into the reality. Since decades, Büchi has supported this philosophy very successfully with new product innovations for evaporation and separation. With the continual development of methods and technologies for the laboratory, we have the pleasure of presenting the revised edition of the «Laboratory Assistant».

This guide will help you acquire a broader and deeper understanding of several of the most widely used laboratory instruments, as rotary evaporators, vacuum pumps, and instruments for melting point determination. The equipment shown in the illustrations are all products of Büchi Labortechnik.

The chapters of the book, have been created in as general terms as possible so that they will also apply to equipment supplied by other manufacturers.

I wish all readers of this «Laboratory Assistant» an interesting time in reading the book, and a challenging and successful work in the laboratory!

Dr. Reinhardt Büchi

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A
**Separation of mixtures
using a rotary evaporator, a
glass oven and a spray dryer**

B
Spray Drying

C
Generation of a vacuum

D
Melting point

Separation of mixtures using a rotary evaporator, a glass oven and a spray dryer



1 Introduction

1.1 Overview

This chapter describes the equipment used for thermal separation of mixtures of materials. The name given to the process differs according to the condition in which the materials being separated are present. Whenever two liquids are being separated, one speaks of distillation. Whenever a liquid is being separated from a solid, one speaks of drying. Sublimation is a separation of solids by evaporating them, bypassing their liquid phase, and then re-solidifying them. Spray drying involves the drying of a solution or a suspension of a compound by spraying it into a hot air stream. Nevertheless, most of these procedures are based on the same principle. This being that different materials have different vapor pressures. The mixture is heated until it evaporates and then is re-condensed. The substance with the lower vapor pressure accumulates in the vapor and in the condensate. There are a great number of devices available to carry out distillation, drying, and sublimation. This chapter restricts itself to the rotary evaporator, glass oven and the spray dryer.

The first section presents the theoretical fundamentals for an understanding of thermal separation processes. The sections that follow deal with the design and applications of the rotary evaporator, the glass oven, the spray dryer and important accessory equipment.

1.2 Areas where rotary evaporators, drying ovens and spray dryers are utilized

Rotary evaporators carry out single distillations and drying quickly and sparingly. The rotation increases the surface area of the mixture, thereby improving the heat transfer. This makes vaporization easier and avoids local overheating and incrustation. It also reduces retarded boiling and foaming. The vacuum lowers the boiling point, making low-temperature distillation possible. In addition, the use of special glass attachments enables carrying out extractions, (re-)crystallizations, and reflux reactions as well as distillation and drying.

Glass ovens are suitable for separating small amounts of mixed substances. They can be used in all separation processes. The possibility of connecting drying ovens to a rotary motor and their vacuum strength make them similar to small rotary evaporators. In addition, they can use bulb-tube distillation to separate multi-component mixtures. Glass ovens are ideal for work being performed on a micro-scale. During the spray drying process a solution or suspension is dispersed into millions of individual droplets with the help of a spray nozzle. The surface area is strongly increased and the solvent, usually water, evaporates in a stream of hot air. Within few seconds the product is present as either a powder, granulate or agglomerate. Spray drying technology is commonly utilized for the reduction of weight and volume in the chemical and foodstuffs industries, as well for the preservation of food.

1.3 Historical development of the rotary evaporator

Distillation, or «drop-by-drop separation», is an art that has been in practice for a long time. The Persians are thought to have invented it in about 3,500 B.C. in order to produce rose water. From Persia, it spread very quickly across the whole of Europe, North Africa, and Asia. In addition to being used for produc-

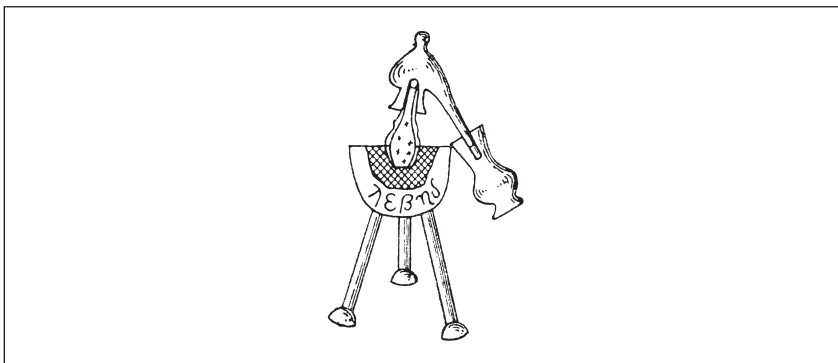


Figure 1:
Historical distillation equipment (2nd century).

ing essences, distillation was also used by mariners for desalinating sea water. Alchemists also practiced distillation. The teachings of alchemy, which arose in Egypt during the 2nd century, represented a combination of religious aspects and chemistry. This was the first «science» that concerned itself in a practical way with material. Aristotelian natural philosophy had postulated the existence of a fundamental material with no qualities, the «prima materia». The alchemists

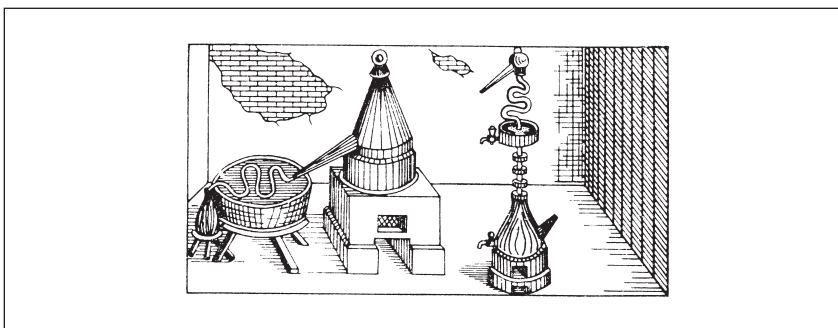


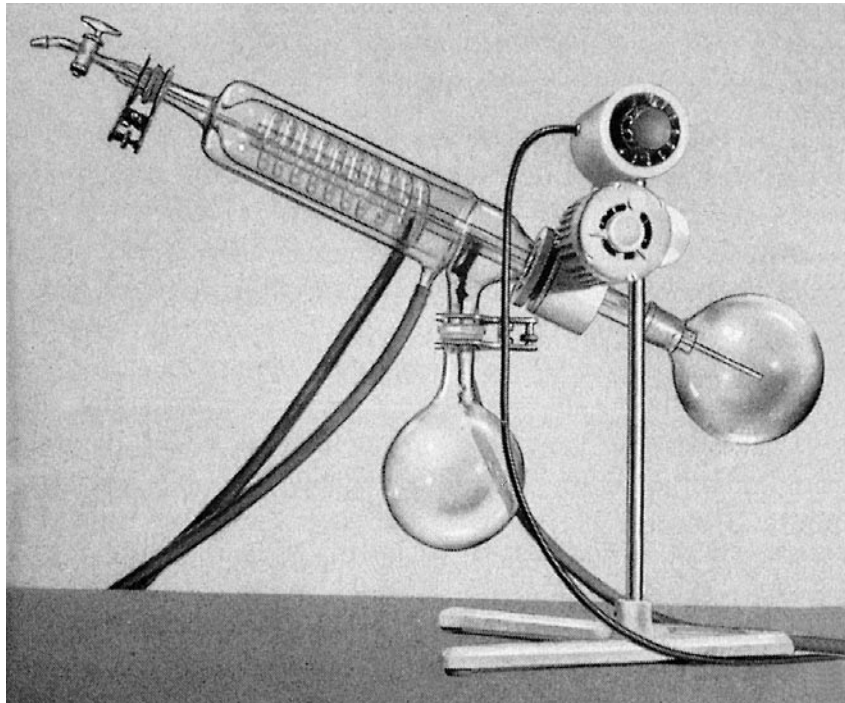
Figure 2:
An air-cooled and a water-cooled distillation device for laboratory use (16th century).

sought to transform natural materials chemically into this basic material, and thento impart to it the new qualities desired, e.g., those of gold (transmutation). In their search they discovered a large number of chemical compounds, improved existing processes and equipment, and discovered new processes that have later been adopted and used in modern chemistry. They also developed distillation apparatus that, from a design-related stand-point, is still being used today. Four classical components of the design include: the heating bath, the bubble flask, the head, and the condenser.

During the 17th and 18th centuries, the focus was on the improvement of existing equipment. The equipment was insulated, the process of continuous distillation was introduced, and water was used as a coolant. The vapor distillation process was also discovered during this time. In selecting the materials for their apparatus, scientists departed more and more from metal and concentrated on glass.

At the end of the 17th century, the Irish physicist Robert Boyle (1627–1691) who had involved himself intensively with vacuums, performed the first vacuum distillations. Finally, during the 19th century, there came the first rectifying columns that made multi-stage distillation possible. A innovative explosion occurred in the mid-19th century with the arrival of organic chemistry. New equipment

Figure 3:
Büchi's first rotary
evaporator.



was specially designed for the needs of the laboratory. Financial involvement by the alcohol industry in France brought about a rapid development on a large industrial scale as well.

The use of a vacuum in distillation processes has played a decisive role ever since Boyle's first experiments. Invention of the pressure regulator and improvement of pumps also enabled a more directed use of the vacuum. In the beginning, only bubble equipment was used for vacuum distillations. Articles by C.C. Draig (1950) and M.E. Volk (1955) published the operating principle behind the rotary evaporator. This process has a far better heat transfer rate than the flask process, which spares the product and increases output. Only two years after Volk's article appeared in early 1957, Büchi Labortechnik in Flawil brought the first rotary evaporator to the market.

For more detailed information on this topic:

– E. Krell: Handbuch der Laboratoriumsdestillation

This book provides an interesting history of distillation. Unfortunately, its presentation concentrates mainly on the development of the equipment and less on the individuals who took part in it.

2 Theoretical basis for distillation

2.1 Thermodynamic basis

2.1.1 Conditions of state, phase transitions

Depending on pressure and temperature, any material may be present in three different phases: solid, liquid, and gaseous. In the solid phase the smallest particles behave as a solid showing only small degrees of motion due to a high binding energy. They swing in a fixed position in their molecular lattices and usually rotate around their axes. The higher the temperature, the more violently they swing or rotate and the distance between the particles usually increases. When heat is applied to a solid material, its kinetic energy increases steadily until, at the melting point, the kinetic energy becomes strong enough to overcome the forces of attraction. The material liquefies. The individual molecules are now able to move about more or less freely within the substance, but the attracting energy between them is still too strong for them to separate from the other molecules. Additional heating of the material removes that energy barrier up to the boiling point. The molecules then have sufficient kinetic energy to enable them to move freely within space without being held back by the forces of attraction. The material is now a gas.

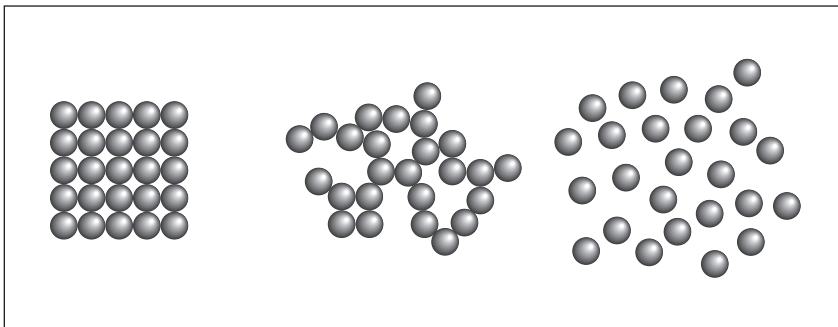


Figure 4:
Particle model of the
3 phases: solid, liquid,
and gas.

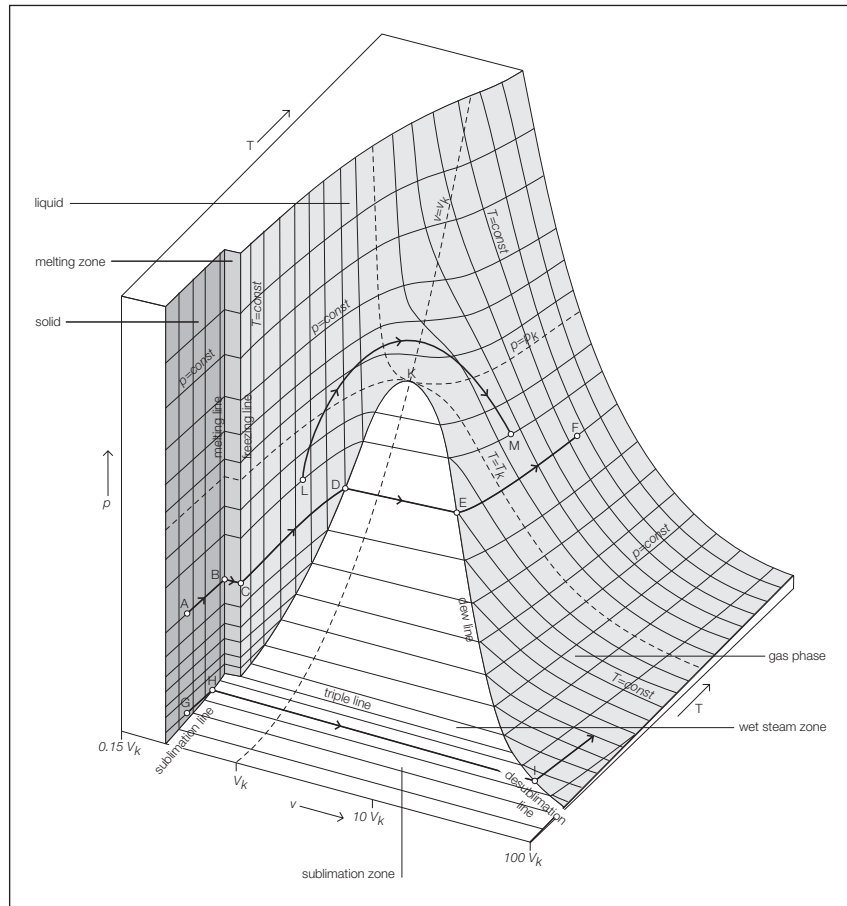
These phase transitions are reversible. When a gas turns into a liquid, people talk of condensation. When a liquid turns into a solid, it freezes or solidifies. If the right conditions are selected, a solid can also be transformed directly into its gaseous phase, bypassing the liquid state. This process, in either direction, is called sublimation. The existing phase is influenced both by the temperature and by the pressure. The lower the pressure, the weaker the forces of attraction between the molecules. This means that both the melting point and the boiling point are lower at low pressures.

A given material may have several solid phases that differ in their crystalline lattice structure but, with the exception of helium, it can have only one liquid and one gaseous phase.

Fig. 5 shows the relationships between temperature, pressure, and volume. This diagram shows various possible paths for going from the solid into the gaseous phase.

- Path from A to F: A solid is being heated under constant atmospheric pressure. When it reaches the melting point, there is a sudden increase in volume: the material liquefies. With further heating, its volume increases steadily until the

Figure 5:
p-V-T surface of a pure material. V has been graphed logarithmically.



temperature reaches the boiling point. The liquid starts to boil, changing into a gas with an enormous increase in its volume. It continues to expand when heated. This is the normal path for a transformation from a solid to a gas.

- Path from G to I: Here the work is being done under reduced pressure. Due to the low pressure, the material begins not to melt but to sublimate. It passes directly into the gas phase.
- Path from L to M: This is a special process. Here the heating is taking place under a higher pressure so that the liquid is transformed into a gas without boiling or evaporating.

Several selected points and zones on this diagram are worth mentioning:

- The triple point: This is the point where a certain pressure and a certain temperature are present at which all three phases are in equilibrium. This combination of values for p and T is referred to as the «triple point».
- The boiling curve / dew point curve: This is that group of combinations for p and T at which the substance begins to boil and/or to condense.
- The melting curve / solidification curve: This is that group of combinations for p and T at which the substance starts to melt or solidify.
- The sublimation line: This is that group of combinations for p and T at which sublimation starts.
- The critical point (K): At pressures and temperatures above this point, no boundaries exist between the gas and liquid phases.

2.1.2 Boiling point, vapor pressure, and evaporation heat

As already shown, a liquid transforms into the gaseous state at its boiling point. This means that no liquid can assume a temperature higher than its boiling point. Experience, of course, tells us that a liquid can also give off gas before it boils: it evaporates at far lower temperatures. When a liquid is evaporating, a few of the molecules on its surface can gather up enough energy to enable them to overcome the forces of attraction and escape into the environment as a gas. This cools the liquid, since an amount of energy is withdrawn from it. The higher the temperature, the more likely it is that a particle will be able to gather up enough energy to escape into the gas phase. The escaping molecules exert a pressure counter to the outside pressure. The more particles that can free themselves from the liquid, the higher that pressure. This vapor pressure depends upon temperature and differs from one compound to the next. It is a measurement for the volatility of a compound. Since the number of particles able to leave the liquid keeps increasing as the temperature keeps rising, vapor pressure rises rapidly with temperature. Once it becomes equal to the outside pressure, there is no longer any force present that can hold the gaseous molecules back in the fluid. The fluid starts to boil.

The Clausius-Clapeyron equation shows how vapor pressure depends on temperature:

$$\frac{d \ln p}{dT} = \frac{L_v}{RT^2} \quad (2.1) \qquad \ln p = -\frac{L_v}{RT} + c \quad (2.2)$$

p is the vapor pressure, T the temperature, R the universal gas constant ($8.3145 \text{ J} \cdot (\text{mol} \cdot \text{K}^{-1})$), and L_v the molar heat of evaporation.

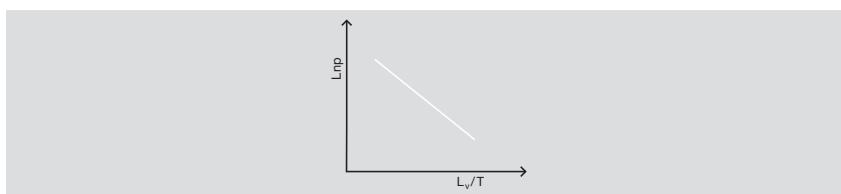


Figure 6:
Logarithmic graph of the
vapor pressure curve.

Shown in the form of a graph, Equation 2.2 appears as the straight line shown in Fig. 6. Its slope depends on the heat of evaporation. Because the heat of evaporation is largely independent of temperature and pressure, it is a constant specific to the given material. Thus, the slopes of the vapor pressure curves for different materials also differ from one another.

The difference in the volatility (vapor pressures) of compounds is the critical factor that makes distillation possible. During the heating process, the more volatile components of a mixture accumulate in the vapor, and thus in its condensate as well. The French chemist François Raoult examined the dependence of vapour pressure on the composition of a component mixture. Through the investigation of mixtures from similar component mixtures (e.g. from benzene and toluene) he found that the quotient p_1/p_1^* is proportional to the mole fraction (x) of solvent 1 in the liquid:

$$\text{Raoult's law: } p_1 = p_1^* \cdot x_1$$

p_1 = Partial vapor pressure
solvent 1
 p_1^* = vapor pressure of pure
solvent 1
 x_1 = mole fraction of
solvent 1 in the liquid

Appendix

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