

Chapter V.I :

Separation by Change of State



Introduction:

Separation by change of state is a fundamental technique used to isolate or purify components of a mixture by exploiting their differences in physical properties, such as boiling point, sublimation point, or miscibility. These methods rely on phase transitions (solid to gas, liquid to gas, etc.) to achieve separation in a controlled manner.

This chapter revisits essential concepts of phase transitions and delves into key separation methods based on changes in state. **Sublimation**, for example, leverages the direct conversion of a solid to a gas, bypassing the liquid phase, and is commonly used for purifying compounds with specific thermal properties. **Distillation**, one of the most widely used techniques, separates liquid mixtures by exploiting differences in boiling points. This chapter explores both simple distillation for components with significant boiling point differences and rectification (fractional distillation) for mixtures with closer boiling points.

Additionally, the chapter addresses the distillation of immiscible liquid mixtures, a method often employed in chemical and food industries to separate compounds that form azeotropes or are difficult to separate by conventional means. These processes not only highlight the elegance of thermodynamic principles but also find critical applications in industries such as petrochemical refining, pharmaceutical production, and environmental engineering.

Through this chapter, students will gain a deeper understanding of the theoretical and practical aspects of separation by change of state, preparing them to apply these techniques effectively in both academic and industrial settings.

VI.1 Separation of Liquid-Liquid Homogeneous Mixtures:

Homogeneous liquid-liquid mixtures occur when two liquids are **completely miscible** or partially miscible. For completely miscible mixtures (e.g., water and ethanol), separation is typically performed by **distillation**. For partially miscible mixtures, **liquid-liquid extraction** is used, a technique based on the differences in solubility of the components in two immiscible solvents.

VI.1.1 Simple Distillation (Separation by State Change):

Simple distillation is a method used to separate liquid components from a mixture by exploiting the differences in their boiling points. This method is effective when the components have a significant boiling point difference (greater than 25°C)

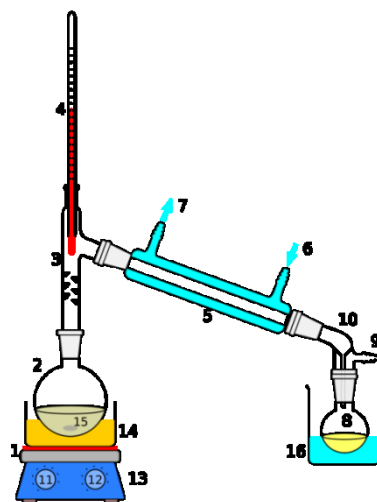


Fig VI.1: Simple Distillation¹

a) Equipment and Apparatus :

- Simple distillation column
- Round-bottom flask
- Thermometer
- Condenser
- Heater
- Receiver (beaker or Erlenmeyer flask)

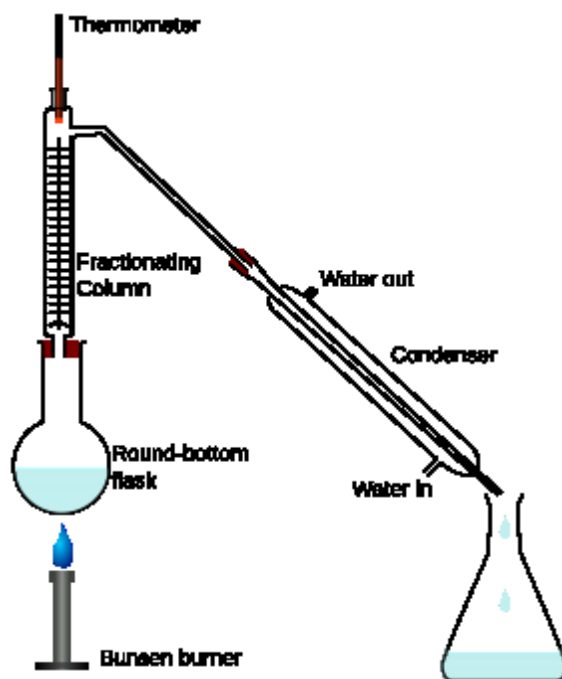
b) Procedure :

- **Heating the mixture:** The liquid mixture is heated in a round-bottom flask.
- **Vaporization:** The component with the lowest boiling point vaporizes first.
- **Condensation:** The vapor rises through the column and passes into a condenser, where it cools and condenses back into liquid form.
- **Collection of distillate:** The condensed liquid is collected in a separate receiver.

VI.1.2 Fractional Distillation:

Fractional distillation is used to separate components of a liquid mixture that have similar boiling points (within 25°C of each other). A fractionating column is used to improve the efficiency of the separation process.

¹ <https://fr.wikipedia.org/wiki/Distillation>



FigVI.2: Fractional Distillation²

c) Equipment and Apparatus :

- Fractionating column (often packed with beads or porous material to increase surface area)
- Round-bottom flask
- Thermometer
- Condenser
- Heater
- Receivers for collecting fractions

d) Procedure :

- **Heating the mixture:** The mixture is heated, causing it to begin vaporizing.
- **Rising vapor:** The vapor rises through the fractionating column, undergoing several cycles of condensation and vaporization.
- **Fractionation:** Through these successive steps, the vapor enriched with the component that has the lowest boiling point reaches the condenser.
- **Condensation and collection:** The different fractions are collected separately as the temperature increases.

Conclusion:

²<https://fr.wikipedia.org/wiki/Distillation>

Fractional distillation provides better separation for mixtures with components that have close boiling points. It is a commonly used technique for separating complex mixtures, such as crude oil.

VI.2 Main separation methods:

In the case of a **solid/liquid mixture**, where the solid is fully dissolved in the liquid, two common methods for separating the solid from the liquid are **crystallization** and **evaporation**. These methods are used when the goal is to recover the solid from the solution or to purify the solid.

A) Crystallization:

Crystallization is a process used to separate a dissolved solid from a liquid by forming solid crystals from the solution. It is based on the difference in solubility of the solid at different temperatures.

B) Evaporation:

Evaporation is a technique used to separate a dissolved solid from a liquid by heating the solution, causing the solvent to vaporize, leaving the solid behind.

Evaporation is often used in industries such as salt production, where the solid (e.g., salt) is left behind after the evaporation of water. It is also commonly used in the food industry to concentrate flavors or ingredients by removing water.

Conclusion:

Evaporation is a straightforward and effective method for recovering dissolved solids from liquids by removing the solvent through heating. It is widely used in applications where the liquid can be discarded or recovered.

Both **crystallization** and **evaporation** are essential techniques for solid/liquid separation. Crystallization is ideal when purification of the solid is desired, while evaporation is used when the focus is on removing the solvent to obtain the solid.

VI.3 Different types of distillation columns and their roles:

- a. Tray (or Stage) Column
- b. Packed Column
- c. Fractionating Column
- d. Reflux Column
- e. Vacuum Distillation Column
- f. Falling Film Distillation Column