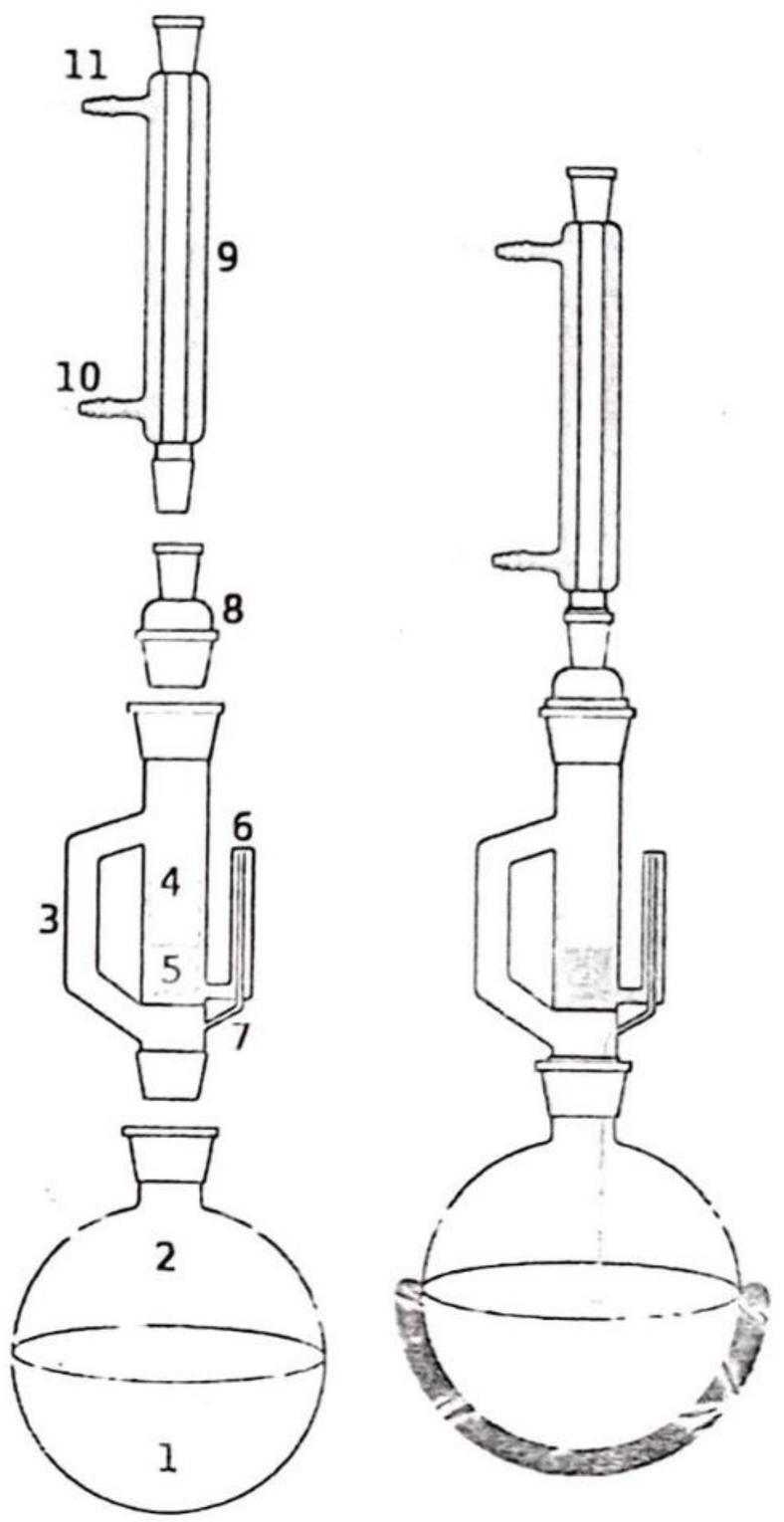
Soxhlet extractor

A **Soxhlet extractor** is a piece of laboratory apparatus[1] invented in 1879 by Franz von Soxhlet.[2] It was originally designed for the extraction of a lipid from a solid material. However, a Soxhlet extractor is not limited to the extraction of lipids. Typically, a Soxhlet extraction is only required where the desired compound has a *limited* solubility in a solvent, and the impurity is insoluble in that solvent. If the desired compound has a significant solubility in a solvent then a simple filtration can be used to separate the compound from the insoluble substance.



A schematic representation of a Soxhlet extractor **1:** Stirrer bar **2:** Still pot (the still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the soxhlet chamber) **3:** Distillation path **4:** Thimble **5:** Solid **6:** Siphon top **7:** Siphon exit **8:** Expansion adapter **9:** Condenser **10:** Cooling water in **11:** Cooling water out

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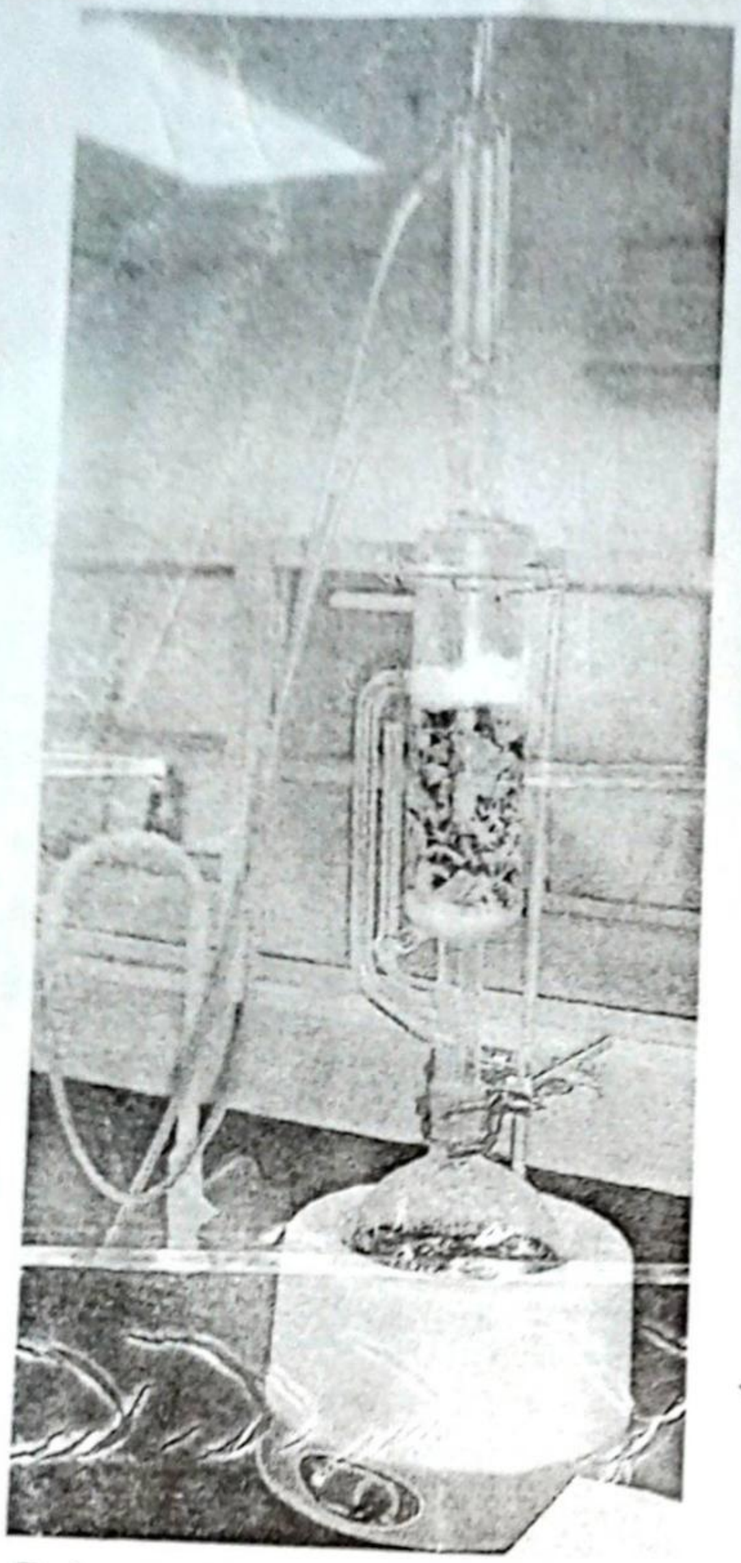


Fruit extraction in progress. The sample is placed in the thimble.

Normally a solid material containing some of the desired compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor is placed onto a flask containing the extraction solvent. The Soxhlet is then equipped with a condenser.

The solvent is heated to reflux. The solvent vapour travels up a distillation arm, and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapour cools, and drips back down into the chamber housing the solid material.

The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days.

During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled.

After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded.

Crystallization

Crystallization is a technique used for the purification of substances. A separation technique to separate solids from a solution.

On adding a solid substance in a liquid and stirring it, the solid dissolves in the fluid. But when added more and more solid to the liquid, a point comes after which no more solid dissolves in the liquid. This point is called saturation point and the fluid is called saturation solution.

Principle:

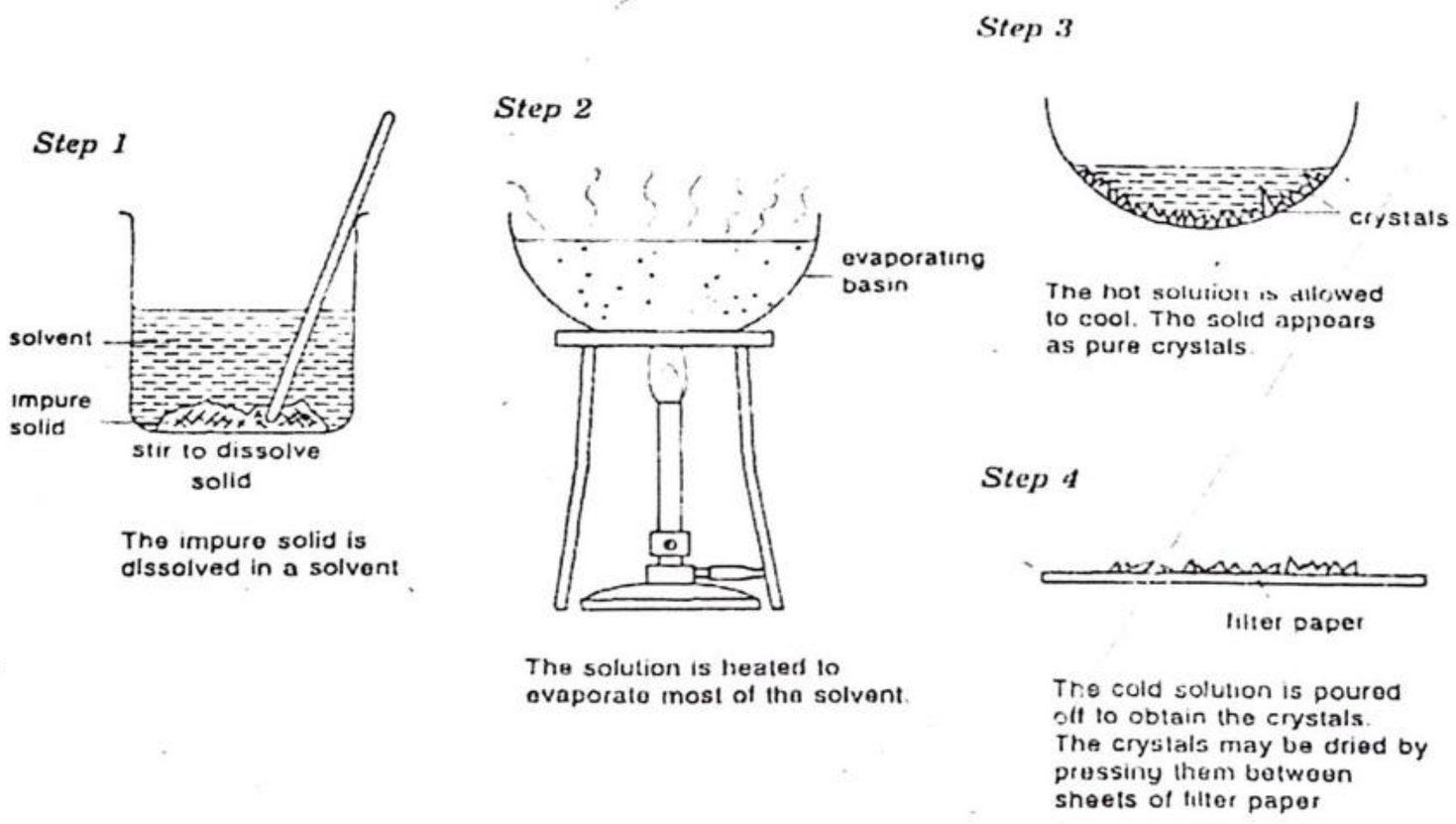
The **principle** behind the **crystallization** is that the amount of solute that can be dissolved by a solvent increases with temperature. In **crystallization**, the impure substance is dissolved in a suitable solvent to reach its nearly saturated solution at a temperature higher than the room temperature.

At this high temperature, the solute has very high solubility in that solvent, so a much smaller quantity of hot solvent is needed for dissolving the solute than the solvent at room temperature. When the solution is cooled, the pure substance is crystallised. The solution left behind is called mother liquor. All the impurities are left behind in the mother liquor. The purification method depends on the differences in solubility between the compound and the impurity.

Steps involved in Crystallization process

1. The solution is heated in an open container
2. The solvent molecules start evaporating, leaving behind the solutes
3. When solution cools, crystals of solute start accumulating on the surface of the solution
4. Crystals are collected and dried as per the product requirement
5. The undissolved solids in the liquid are separated by the process of filtration
6. The size of crystals formed during this process depends on the cooling rate.
7. Large number of tiny crystals are formed, if the solution is cooled at a fast rate
8. Large crystals are formed at slow cooling rates

→ nucleation



Separation technique of substance by Crystallization

Sublimation

Sublimation is a process of a solid getting directly converted into its vapour on heating without becoming a liquid. This happens below the melting point of the solid. The vapour on condensation gives back the solid. Sublimation is an alternative to crystallization for the purification of some solids. The differing vapour pressures of the substance which sublimes and the impurities help in purifying a substance using sublimation.

Principle

The process of sublimation can be understood by studying the pressure-temperature phase diagram for a substance which sublimes (Fig. 6.9). The figure relates the solid, liquid and vapour states of the substance with pressure and temperature. It indicates that the liquid state cannot exist with conditions of temperature and pressure below those depicted at point *O* since the liquid state is thermodynamically unstable. The vapour pressure of the solid at any temperature below the melting point is given by the curve *OA*, This curve represents the equilibrium between the solid and vapour, which is of importance for sublimation. The point *O* is called the triple point and if the temperature of the vapour at a pressure below the triple point is reduced, the vapour will directly condense to form a solid.

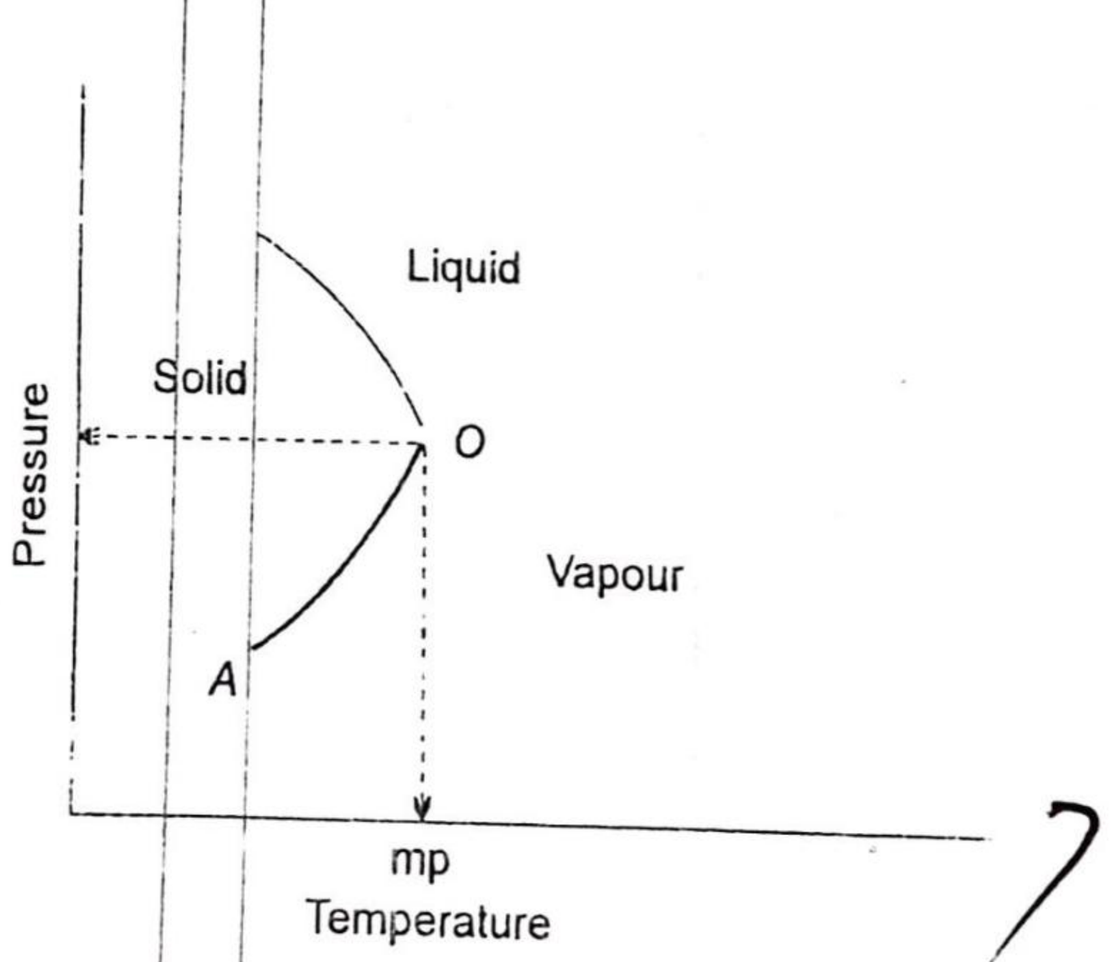


Fig. 6.9 Phase Diagram for Sublimination

At the triple point solid, liquid and vapour co-exist. In order that a solid may pass directly into vapour without the intermediate formation of a liquid phase, the pressure of the vapour

must not be allowed to exceed that of the triple point. This can be done if the vapour pressure at the triple point is fairly high and consequently the rate of vaporization will be considerable. Under these conditions, purification of the solid by sublimation at atmospheric pressure is possible.

Example

Camphor has a triple point at 179° C and an equilibrium pressure of 370 mm. When it is slowly heated below 179°, it will vaporize without melting and if the vapour is deposited on a cold surface, the pressure will be kept below 370 mm and hence vaporization will continue until all the solid has disappeared. Condensation occurs on the cold surface directly because the pressure is below that at the triple point.

Technique of Sublimation

A simple form of apparatus for sublimation is shown in Fig. 6.10. The substance to be sublimed is placed in an evaporating dish. A narrow ring of pyrex glass is fitted near the rim, which supports a filter paper with a number of small holes made in an upward direction. A funnel with a plug of glass wool in the stem is kept inverted over the paper. The dish is gently heated. Vapours escape through the holes in the paper and condense on the upper surface of the filter paper and also on the walls of the funnel. Heating is stopped when most of the material in the dish has vaporised. Supply of heat should be such that the funnel does not become hot. The rate of sublimation may be increased by applying very gentle suction at the stem of the funnel. This will draw the vapour into the condensing chamber.

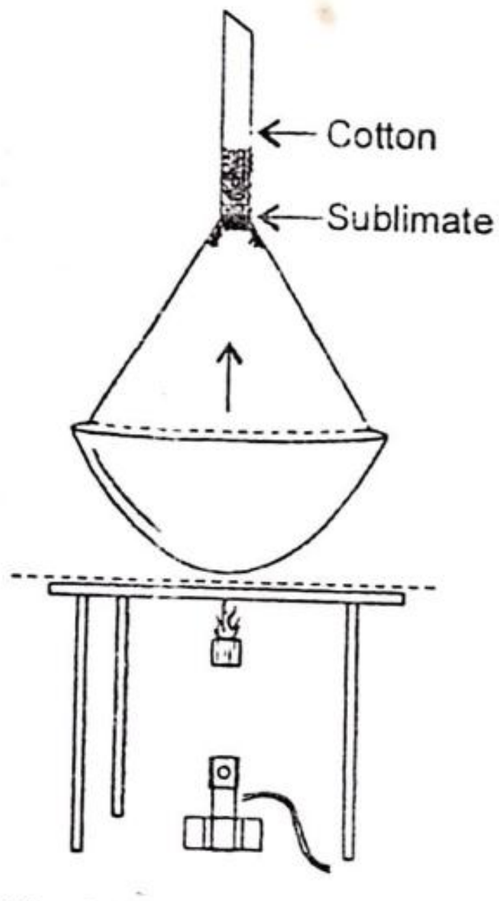


Fig. 6.10 Simple Sublimation Apparatus

Types of Sublimation

1. *Simple Sublimation.* A simple sublimation is conducted at atmospheric pressure. For example, hexachloroethane attains a vapour pressure of 760 mm below its melting point. Attempt to heat solid hexachloroethane above 180°C (760 mm) in an unstoppered vessel will cause an outgoing of the vapour and the whole material will sublime and escape into the atmosphere.

2. *Sublimation Under Reduced Pressure.* This can be carried out by using the apparatus shown in Fig. 6.11. The cold finger is fitted into the larger tube by means of rubber stopper and carries a disc slightly smaller in diameter than the outer tube. The pressure can be reduced by a water or an oil pump. The impure material is taken in the wider tube. On heating gently, either with a small flame or by an oil bath, the substance sublimes and collects on the cold surface of the disc.

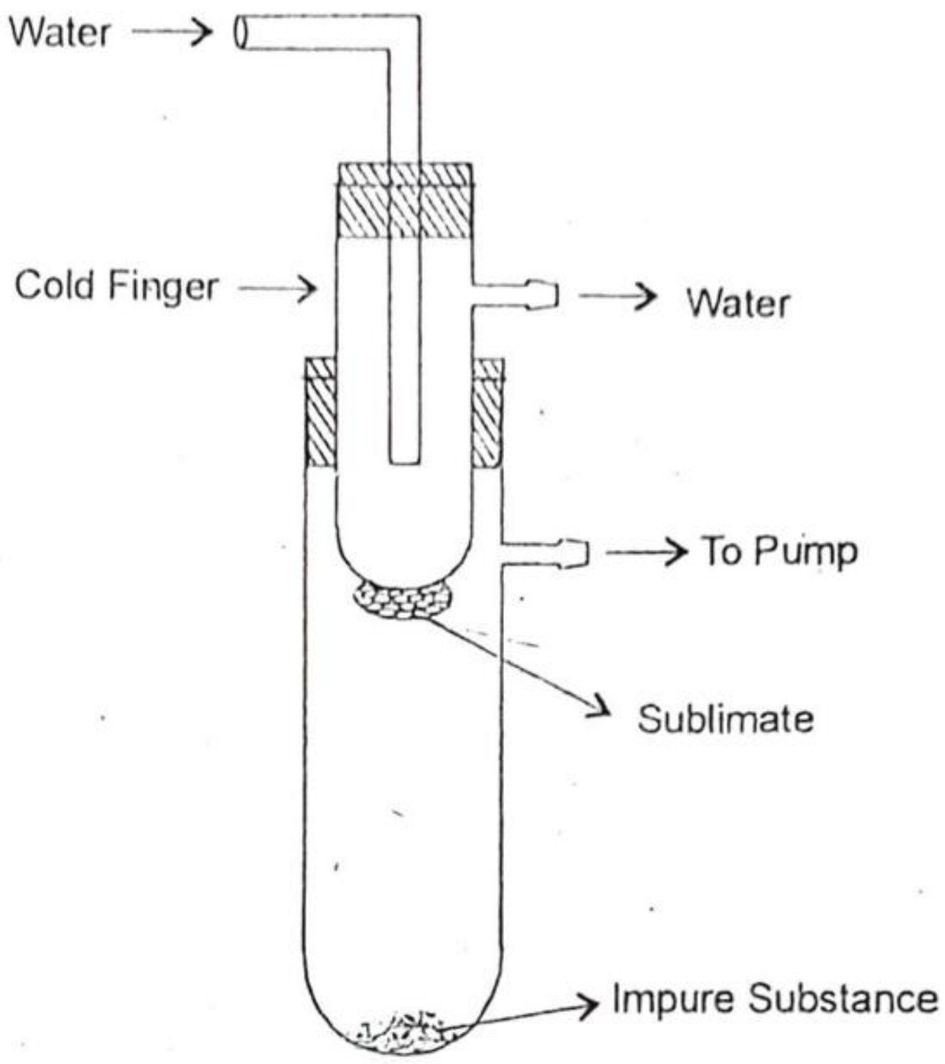


Fig. 6.11 Sublimation under Reduced Pressure

3. *Vacuum Sublimation.* Very few organic compounds exhibit vapour pressure adequate for sublimation at atmospheric pressure. Reduced pressure (vacuum) is needed to increase the rate of evaporation of the solid. This method is similar to the use of vacuum distillation for high boiling liquids. Most solids melt at temperatures below those at which their vapour pressures reach 760 mm. Naphthalene and phthalic anhydride belong to this type. These are sublimed readily under reduced pressure and this technique is called vacuum sublimation. Sublimation under high vacuum is effective for separating a substance from a mixture.

4. *Entrainer Sublimation.* This is a technique of subliming a substance (which must otherwise be sublimed under vacuum) at atmospheric pressure below the melting point by introducing an inert gas as an entrainer. The vapour of the substance is swept by the inert gas into a condenser where it crystallizes.

Advantages of Sublimation

1. Sublimation is a convenient and rapid method for purification of solids when impurities are either non-volatile or considerably more volatile than the desired compound.
2. In distillation, volatile impurities tend to dissolve in the newly formed distillate as the distillate is formed in the condenser; in sublimation this is impossible as crystals are formed directly from the vapour, the impurity molecules still remaining as vapour.
3. The apparatus required for sublimation is simple and inexpensive.

Sublimation is restricted to relatively non-polar substances having symmetrical structures. In these cases, crystal forces are weaker and vapour pressures are higher. These properties increase the ease with which a molecule can escape from the solid to the vapour phase. Weak intermolecular attractive forces between the molecules of the solid promote easy sublimation. In a symmetrical structure, the electron density is symmetrically distributed and as such dipole moments are smaller and intermolecular attractions are weaker. A smaller dipole moment implies a higher vapour pressure. The van der Waals forces also influence the ease of sublimation, but to a lesser extent compared to electrostatic forces. When the van der Waals forces are of high magnitude as with large molecules, the substance is less likely to sublime.

Zone Refining

This is a process for the purification of certain solid materials. In this process, the impure solid material in the form of rod is slowly passed through a small heated zone (Fig. 6.12). The temperature of this zone is kept above the melting point of the solid material using an electrical heating coil. The impurities (solutes) dissolve in the molten metal (solvent). When the rod emerges from the heating coil, it gets cold and the pure metal crystallizes in the cold emerging rod and the impurities migrate to the hot molten zone of the rod which is still in the heating coil. The metal crystal lattice formed on cooling does not accommodate the impurities. The process of heating and cooling is repeated due to which all the impurities reach one end (hot) of the rod. Then this impure end is cut off and discarded; the remaining rod is of pure metal.

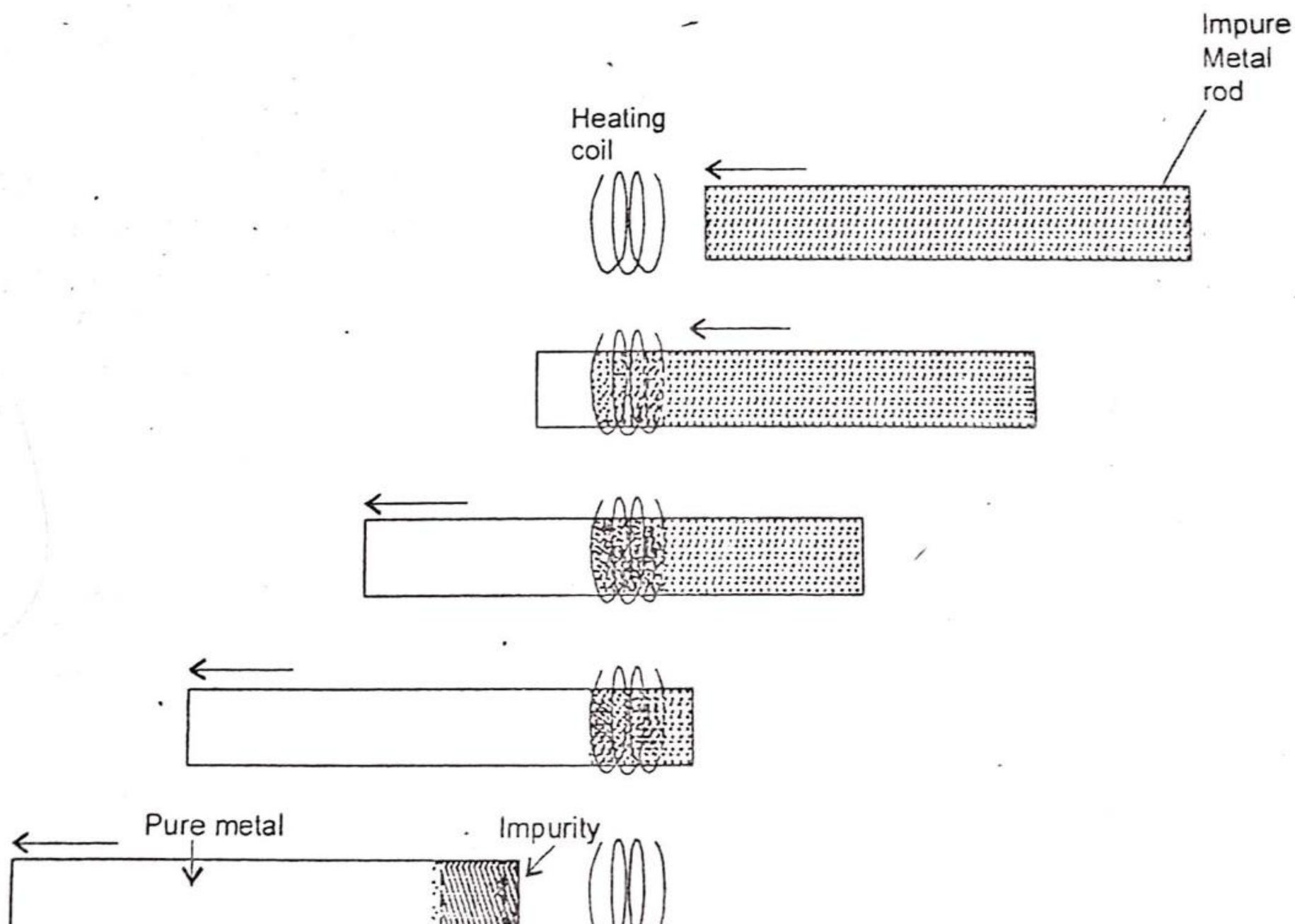


Fig. 6.12 Zone-refining Technique for Purifying Metals

This method is actually *fractional crystallization*; the impurities tend to remain dissolved in the molten metal rather than in the solid metal. *Zone melting* is another name for this process.