

Quadrant II – Transcript and Related Materials

Programme: Bachelor of Science (First Year)

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Unit: I

**Module Name: Different techniques in Chemistry and their Applications:
Distillation, Recrystallisation**

Module No: 01

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Notes:

A large number of methods are available for the purification of substances. The choice of method, however, depends upon the nature of substance (whether solid or liquid). It also depends on the type of impurities present in it. We commonly use these methods for purification of substances: Recrystallisation, Fractional crystallisation, Sublimation, Simple distillation, Fractional distillation, Distillation under reduced pressure, Steam distillation, Azeotropic distillation, Chromatography.

An important organic process used to separate two or more than two liquids having different boiling points from a liquid mixture.(based on volatilities). The liquid to be distilled is vaporized and the vapors are subsequently channeled into a condenser that cools the vapors to obtain a pure liquid. According to the differences in boiling points between the liquids, distillation process can be classified into different types:-

1-Simple distillation

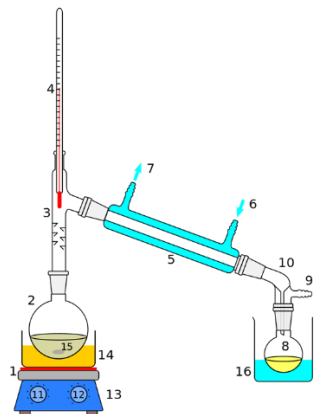
3-Steam distillation.

2-Fractional distillation

4-Vacuum distillation.

1) Simple distillation -

Simple distillation is usually used only to separate liquids whose boiling points differ greatly ($>25^{\circ}\text{C}$). Thus, a mixture of two liquids, volatile and non-volatile, is heated in the boiling assembly and only the liquid with low boiling point distills out. In this case, the distillate may be sufficiently pure for its intended purpose. Distillation using air condenser is used for liquids with boiling points higher than 100°C .



Fractional distillation

- Those mixtures, in which the volatility of the components is nearly similar or differs by 25°C (at 1 atmosphere pressure), cannot be separated by simple distillation.
- In such cases, fractional distillation is used whereby the constituents are separated by a fractionating column.
- In the fractionating column, the plates are arranged and the compound with the least boiling point are collected at the top while those with higher boiling point are present at the bottom.
- A series of compounds are separated simultaneously one after another. Fractional distillation is used for the alcohol purification and gasoline purification in petroleum refining industries.

Steam distillation

- Steam distillation is used for the purification of mixtures, in which the components are temperature or heat sensitive; for example, organic compounds.

- In the instrument setup, steam is introduced by heating water, which allows the compounds to boil at a lower temperature.
- This way, the temperature sensitive compounds are separated before decomposition.
- The vapors are collected and condensed in the same way as other distillation types.
- The resultant liquid consists of two phases, water and compound, which is then purified by using simple distillation.
- Steam distillation is practiced for the large-scale separation of essential oils and perfumes.

Vacuum distillation (distillation under reduced pressure)

- Vacuum distillation is a special method of separating compounds at pressure lower than the standard atmospheric pressure.
- Under this condition, the compounds boil below their normal boiling temperature. Hence, vacuum distillation is best suited for separation of compounds with higher boiling points (more than 200°C), which tend to decompose at their boiling temperature.
- Vacuum distillation can be conducted without heating the mixture, as usually followed in other distillation types.
- For the separation of some aromatic compounds, vacuum distillation is used along with steam distillation.

Recrystallisation-

Solid organic compounds when isolated from organic reactions are seldom pure, they are usually contaminated with small amounts of other compounds (impurities) which are produced along with the desired product. The purification of impure crystalline compounds is usually effected by crystallization from a suitable solvent or mixture of solvents. The purification of solids by crystallization is based upon differences in their solubility in a given solvent or mixture of solvents.

In its simplest form, the crystallization process consist of dissolving the impure substance in some suitable solvent at or near the boiling point, filtering the hot solution from particles of insoluble material and dust, Allowing the hot solution

to cool thus causing the dissolved substance to crystallise out; Separating the crystals from the supernatant solution (mother-liquor)

The resultant solid, after drying is tested for purity, usually melting point determination, spectroscopic methods, TLC and if found impure is again recrystallized from fresh solvent.

The most desirable characteristics of a solvent for recrystallisation are as follows:

A high solvent power for the substance to be purified at high temperature and a comparatively low solvent power at the laboratory temperature or below.

- It should dissolve the impurities readily or to only a very small extent
- It should yield well- formed crystals of the purified compound.
- It must be capable of easy removal from the crystals of the purified compound,(possess a relatively low boiling point)
- Common solvents for recrystallisation are distilled water, methanol, ethanol, acetone, ethyl acetate, also solvent pairs such as alcohol & water or alcohol and toluene can be used.

APPLICATIONS OF PURIFICATION TECHNIQUES –

- 1) Sugar having an impurity of common salt can be crystallized from hot ethanol since sugar dissolves in hot ethanol but common salt does not.
- 2) The drug Stavudine, which is used to reduce the effects of HIV, is typically isolated by crystallization.
- 3) Fractional distillation is employed for refining of petroleum products on a large scale
- 4) Distillation permits separation of air into its components — notably oxygen, nitrogen, and argon — for industrial use.
- 5) Distillation of fermented products produces distilled beverages with a high alcohol content, or separates out other fermentation products of commercial value.