

Organic Chemistry: Basic Principles & Techniques

Section 8.8: Methods of Purification



Purification is the absolute prerequisite for chemical analysis.

Natural extraction and laboratory synthesis rarely yield pure organic compounds. Before any structural investigation can begin, a compound must be isolated from its crude matrix.

The Metric of Purity

Historically, purity is verified by observing sharp physical constants:

- Sharp melting point (for solids)
- Sharp boiling point (for liquids)

The Purification Sequence

Crude Extraction
(Contains impurities)



Purification Technique
(Exploits a specific property difference)



Pure Compound
(Yields sharp mp/bp)

Sublimation separates solids by exploiting extreme vapor pressure differences.



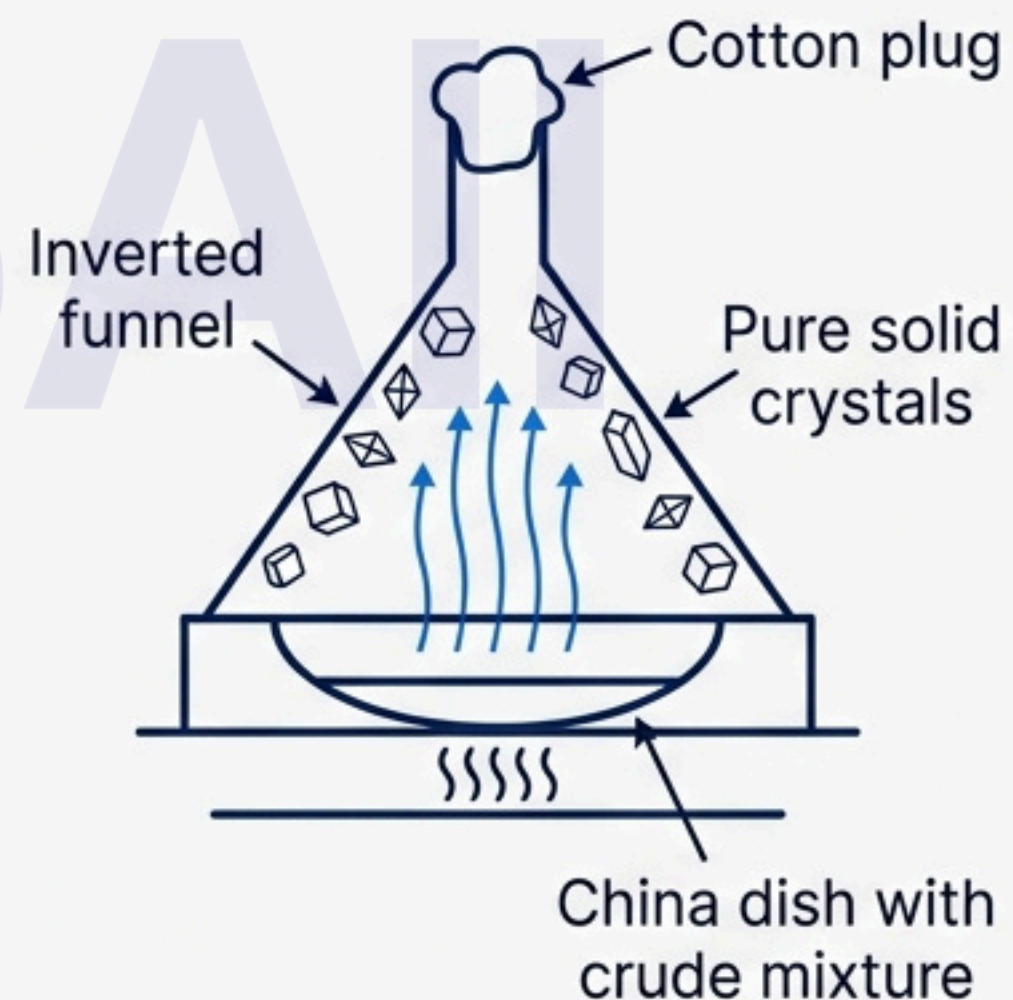
Principle

Direct transition from solid to vapor state, completely bypassing the liquid phase upon heating.

Diagnostic Condition

The mixture must contain **one sublimable organic compound** mixed with **non-sublimable impurities**.

Visual Model



Crystallisation isolates compounds via temperature-dependent solubility.

Based on differences in solubility profiles in a suitable solvent. The organic compound must be **sparingly soluble at room temperature** but **highly soluble at elevated temperatures**.



1. Dissolution

Dissolve the crude compound in a minimum volume of a suitable solvent at its boiling point.



2. Hot Filtration

Filter hot to remove impurities that are insoluble in the solvent at high temperatures.



4. Separation

Filter cold to isolate pure crystals. Soluble impurities remain trapped in the mother liquor.



3. Cooling

Cool the concentrated hot filtrate slowly to induce crystallization of the pure compound.



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Distillation separates volatile liquids based on boiling points and thermal stability.



Simple Distillation

Condition: Liquids with widely different boiling points ($\Delta bp > 40^\circ\text{C}$) or separating volatile liquids from non-volatile impurities.

Mechanism: Vaporization followed by condensation.

Example: Chloroform (bp 334K) and Aniline (bp 457K).

Fractional Distillation

Condition: Liquids with very close boiling points.

Mechanism: Uses a fractionating column. Vapors undergo a series of successive condensations and vaporizations across theoretical plates.

Example: Crude oil components in the petroleum industry.

Reduced Pressure (Vacuum)

Condition: Liquids that have very high boiling points OR decompose at or below their normal boiling points.

Mechanism: Pressure is reduced so the liquid boils at a lower temperature, preventing decomposition.

Example: Glycerol from spent-lye in the soap industry.

Steam Distillation

Condition: Substances that are steam volatile but immiscible with water.

Mechanism: $P_{\text{total}} = P_{\text{org}} + P_{\text{water}}$. Organic liquid boils at a lower temperature than its normal bp because $P_{\text{org}} < P_{\text{total}}$.

Example: Aniline-water mixture.

Extraction and chromatography rely on differential phase affinity.



Differential Extraction

Principle: Separating an organic compound from its aqueous solution by shaking it with an immiscible organic solvent.

Condition: The organic compound must be more soluble in the organic solvent than in water.

Setup: Performed in a separatory funnel. The organic layer (now containing the compound) is separated, and the solvent is distilled off.

(Note: If solubility is very low, continuous extraction is utilized).

Chromatography

Principle: Differential movement or affinity of individual components between a stationary phase and a mobile phase.

Categories:

1. **Adsorption Chromatography:** Components adsorb to different degrees on a solid stationary phase (e.g., Column Chromatography, Thin Layer/TLC). Analyzed via Retardation factor (R_f).
2. **Partition Chromatography:** Continuous differential partitioning of components between a stationary liquid and a mobile phase (e.g., Paper Chromatography).

Diagnostic Matrix: Map the property difference directly to the purification method.

Property Difference Identified in Mixture	Recommended Purification Method	Key Example / Note
Vapor Pressure: One solid sublimable, one not	Sublimation	Camphor, Naphthalene separation
Solubility (Temp): High at bp, low at room temp	Crystallisation	Uses hot filtration to remove insolubles
Boiling Point: Large difference ($\Delta bp > 40^{\circ}\text{C}$)	Simple Distillation	Chloroform (334K) & Aniline (457K)
Boiling Point: Very close boiling points	Fractional Distillation	Crude oil refining (uses fractionating column)
Stability: Decomposes at or below boiling point	Reduced Pressure Distillation	Glycerol from spent-lye
Volatility: Steam volatile & water immiscible	Steam Distillation	$P_{\text{total}} = P_{\text{org}} + P_{\text{water}}$
Solubility (Solvent): Higher affinity for organic phase	Differential Extraction	Uses separatory funnel
Phase Affinity: Differential adsorption/partitioning	Chromatography	TLC (R_f value), Column, Paper



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