

PRACTICAL ORGANIC CHEMISTRY

PURIFICATION METHODS DISTILLATION TECHNIQUES

Type :

(A) SIMPLE DISTILLATION

Conditions

- (i) When liquid sample has non-volatile impurities
- (ii) When boiling point difference is 30 K or more.

Examples

- (i) Mixture of chloroform (BP = 334K) and Aniline (BP = 457K)
- (ii) Mixture of Ether (b.p. = 308K) & Toluene (b.p. = 384K)
- (iii) Hexane (342K) and Toluene (384K)

(B) FRACTIONAL DISTILLATION

When b.p. difference is 10K

Examples

- (i) Crude oil in petroleum industry
- (ii) Acetone (329 K) and Methyl alcohol (338K)

(C) DISTILLATION UNDER REDUCED PRESSURE

(Vacuum distillation)

When liquid boils at higher temperature and it may decompose before b.p. is attained.

Example

- (i) Concentration of sugar juice
- (ii) Recovery of glycerol from spent lye.
- (iii) Glycerol

(D) STEAM DISTILLATION

When the substance is immiscible with water and steam volatile.

Example :

- (i) Aniline is separated from water
- (ii) Turpentine oil
- (iii) Nitro Benzene
- (iv) Bromo Benzene
- (v) Naphthalene
- (vi) o-Nitrophenol

$$P = P_1 + P_2$$

Vapour pressure of Organic liquid

Vapour pressure of water

LASSAIGNE'S METHOD (detection of elements)

Element	Sodium extract	Confirmed test
Nitrogen	$\text{Na} + \text{C} + \text{N} \xrightarrow{\Delta} \text{NaCN}$	$(\text{NaCN} + \text{FeSO}_4 + \text{NaOH}) \xrightarrow{\text{boil and cool}} \text{Fe}_3(\text{Fe}(\text{CN})_6)_2$ Prussian blue colour
Sulphur	$2\text{Na} + \text{S} \xrightarrow{\Delta} \text{Na}_2\text{S}$	(i) $\text{Na}_2\text{S} + \text{Na}_2(\text{Fe}(\text{CN})_6)\text{NO}_3 \xrightarrow{\text{Boil in HCl}} \text{Na}_2(\text{Fe}(\text{CN})_6)\text{N}_2\text{O}_5$ a deep violet colour (ii) $\text{Na}_2\text{S} + \text{CH}_3\text{COOH} \rightarrow \text{CH}_3\text{COONa} + \text{H}_2\text{S}$ A black ppt. (PbS)
Halogen	$\text{Na} + \text{X} \xrightarrow{\Delta} \text{NaX}$	$\text{NaX} + \text{HNO}_3 \rightarrow \text{AgNO}_3$ (i) White ppt. soluble in aq. NH_3 , confirms Cl. (ii) Yellow ppt. partially soluble in aq. NH_3 , confirms Br. (iii) Yellow ppt. insoluble in aq. NH_3 , confirms I.
Nitrogen and sulphur together	$\text{Na} + \text{C} + \text{N} + \text{S} \xrightarrow{\Delta} \text{NaCNS}$ Sodium thiocyanate (Blood red colour)	As in test for nitrogen; instead of green or blue colour, blood red colouration confirms presence of N and S both.

QUANTITATIVE ANALYSIS OF ORGANIC COMPOUNDS

Estimation of carbon and hydrogen - Leebly's method



$$\% \text{ of C} = \frac{12}{44} \times \frac{\text{wt. of CO}_2}{\text{wt. of org. compd.}} \times 100$$

$$\% \text{ of H} = \frac{2}{18} \times \frac{\text{wt. of H}_2\text{O}}{\text{wt. of org. compd.}} \times 100$$

Note : This method is suitable for estimation if organic compound contains C and H only. In case if other elements e.g. N, S, halogens are also present the organic compound will also give their oxides which is being absorbed in KOH and will increase the percentage of carbon and therefore following modification should be made.

ESTIMATION OF NITROGEN

Duma's method :

The nitrogen containing organic compound yields nitrogen gas on heating it with copper (II) oxide in the presence of CO_2 gas. The mixture of gases is collected over potassium hydroxide solution in which CO_2 is absorbed and volume of nitrogen gas is determined.

$$\% \text{ of N} = \frac{28}{22400} \times \left(\frac{\text{Vol. of N}_2 \text{ collected at N.T.P.}}{\text{Vol. of organic compound}} \right) \times 100$$

Note : This method can be used to estimate nitrogen in all types of organic compounds

Kjeldahl's method :

In this method nitrogen containing compound is heated with conc. H_2SO_4 in presence of copper sulphate to convert nitrogen into ammonium sulphate which is decomposed with excess of alkali to liberate ammonia. The ammonia evolved is

$$\% \text{ of N} = \frac{1.4 \times \text{volume of acid (ml)} \times \text{normality of acid}}{\text{wt of organic compound}}$$

Note : This method is simpler and more convenient and is mainly used for finding out the percentage of nitrogen in food stuffs, soil, fertilizers and various agricultural products. This method cannot be used for compound having nitro groups, azo group ($-\text{N}=\text{N}-$) and nitrogen in the ring (pyridine, quinoline etc.) Since nitrogen in these compounds is not quantitatively converted in to ammonium sulphate.

Msc Organic Chemistry Practical Manual

R Barnett



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