

# 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                      | $\text{Na} + \text{C} + \text{N} \xrightarrow{\Delta} \text{NaCN}$                                                      | $(\text{NaCN} + \text{FeSO}_4 + \text{NaOH}) \xrightarrow{\text{boil and cool}} \text{Fe}_2(\text{Fe}(\text{CN})_6)_3$<br>Prussian blue colour                                                                                                                                                                                     |
| Sulphur                       | $2\text{Na} + \text{S} \xrightarrow{\Delta} \text{Na}_2\text{S}$                                                        | (i) $\text{Na}_2\text{S} + \text{Na}_2(\text{Fe}(\text{CN})_6)\text{NO}_3 \xrightarrow{\text{Boil in HCl}} \text{Na}_2(\text{Fe}(\text{CN})_6)\text{N}_2\text{O}_5$<br>a deep violet colour<br>(ii) $\text{Na}_2\text{S} + \text{CH}_3\text{COOH} \rightarrow (\text{CH}_3\text{COO})_2\text{S} \rightarrow$<br>A black ppt. (PbS) |
| Halogen                       | $\text{Na} + \text{X} \xrightarrow{\Delta} \text{NaX}$                                                                  | $\text{NaX} + \text{HNO}_3 \rightarrow \text{AgNO}_3$<br>(i) White ppt. soluble in aq. $\text{NH}_3$ , confirms Cl.<br>(ii) Yellow ppt. partially soluble in aq. $\text{NH}_3$ , confirms Br.<br>(iii) Yellow ppt. insoluble in aq. $\text{NH}_3$ , confirms I.                                                                    |
| Nitrogen and sulphur together | $\text{Na} + \text{C} + \text{N} + \text{S} \xrightarrow{\Delta} \text{NaCNS}$<br>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  $\text{CO}_2$  gas. The mixture of gases is collected over potassium hydroxide solution in which  $\text{CO}_2$  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.  $\text{H}_2\text{SO}_4$  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

**L Cohen**



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**Table of Contents Msc Organic Chemistry Practical Manual**

1. Understanding the eBook Msc Organic Chemistry Practical Manual
  - The Rise of Digital Reading Msc Organic Chemistry Practical Manual
  - Advantages of eBooks Over Traditional Books
2. Identifying Msc Organic Chemistry Practical Manual
  - Exploring Different Genres
  - Considering Fiction vs. Non-Fiction
  - Determining Your Reading Goals
3. Choosing the Right eBook Platform
  - Popular eBook Platforms
  - Features to Look for in an Msc Organic Chemistry Practical Manual
  - User-Friendly Interface
4. Exploring eBook Recommendations from Msc Organic Chemistry Practical Manual
  - Personalized Recommendations
  - Msc Organic Chemistry Practical Manual User Reviews and Ratings
  - Msc Organic Chemistry Practical Manual and Bestseller Lists
5. Accessing Msc Organic Chemistry Practical Manual Free and Paid eBooks
  - Msc Organic Chemistry Practical Manual Public Domain eBooks
  - Msc Organic Chemistry Practical Manual eBook Subscription Services
  - Msc Organic Chemistry Practical Manual Budget-Friendly Options
6. Navigating Msc Organic Chemistry Practical Manual eBook Formats
  - ePub, PDF, MOBI, and More
  - Msc Organic Chemistry Practical Manual Compatibility with Devices
  - Msc Organic Chemistry Practical Manual Enhanced eBook Features
7. Enhancing Your Reading Experience
  - Adjustable Fonts and Text Sizes of Msc Organic Chemistry Practical Manual
  - Highlighting and Note-Taking Msc Organic Chemistry Practical Manual
  - Interactive Elements Msc Organic Chemistry Practical Manual
8. Staying Engaged with Msc Organic Chemistry Practical Manual

- Joining Online Reading Communities
- Participating in Virtual Book Clubs
- Following Authors and Publishers Msc Organic Chemistry Practical Manual
- 9. Balancing eBooks and Physical Books Msc Organic Chemistry Practical Manual
  - Benefits of a Digital Library
  - Creating a Diverse Reading Collection Msc Organic Chemistry Practical Manual
- 10. Overcoming Reading Challenges
  - Dealing with Digital Eye Strain
  - Minimizing Distractions
  - Managing Screen Time
- 11. Cultivating a Reading Routine Msc Organic Chemistry Practical Manual
  - Setting Reading Goals Msc Organic Chemistry Practical Manual
  - Carving Out Dedicated Reading Time
- 12. Sourcing Reliable Information of Msc Organic Chemistry Practical Manual
  - Fact-Checking eBook Content of Msc Organic Chemistry Practical Manual
  - Distinguishing Credible Sources
- 13. Promoting Lifelong Learning
  - Utilizing eBooks for Skill Development
  - Exploring Educational eBooks
- 14. Embracing eBook Trends
  - Integration of Multimedia Elements
  - Interactive and Gamified eBooks

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