

# PRACTICAL ORGANIC CHEMISTRY

## PURIFICATION METHODS DISTILLATION TECHNIQUES

Type :

### (A) SIMPLE DISTILLATION

#### Conditions

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

#### Examples

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

### (B) FRACTIONAL DISTILLATION

When b.p. difference is 10K.

#### Examples

- Crude oil in petroleum industry
- 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 :**
- Concentration of sugar juice
  - Recovery of glycerol from spent lye.
  - Glycerol

### (D) STEAM DISTILLATION

When the substance is immiscible with water and steam volatile.

#### Example :

- Aniline is separated from water
- Turpentine oil
- Bromo Benzene
- o-Nitrophenol
- Nitro Benzene
- Naphthalene

$$P = P_s + P_w$$

Vapour pressure of Organic liquid = Vapour pressure of water

## LASSAIGNE'S METHOD (detection of elements)

Element	Sodium extract	Confirmed test
Nitrogen	$\text{Na} + \text{C} + \text{N} \xrightarrow{\Delta} \text{NaCN}$	$\text{NaCN} + \text{FeSO}_4 + \text{NaOH} \xrightarrow{\text{boil and cool}} \text{FeCl}_2 + \text{conc. HCl} \rightarrow \text{Fe}_2(\text{Fe}(\text{CN})_6)_3$ <p>Prussian blue colour</p>
Sulphur	$2\text{Na} + \text{S} \xrightarrow{\Delta} \text{Na}_2\text{S}$	<ol style="list-style-type: none"> <li><math>\text{Na}_2\text{S} + \text{Na}_2(\text{Fe}(\text{CN})_6)\text{NO} \xrightarrow{\text{Boil in benzene}} \text{Na}_2(\text{Fe}(\text{CN})_6)\text{NO.S}</math> a deep red colour</li> <li><math>\text{Na}_2\text{S} + \text{CH}_3\text{COOH} + \text{CH}_3\text{COOLi} \rightarrow \text{A black ppt. (LiS)}</math></li> </ol>
Halogen	$\text{Na} + \text{X} \xrightarrow{\Delta} \text{NaX}$	<ol style="list-style-type: none"> <li><math>\text{NaX} + \text{HNO}_3 + \text{AgNO}_3</math></li> <li>White ppt. soluble in aq. <math>\text{NH}_3</math>, confirms Cl.</li> <li>Yellow ppt. partially soluble in aq. <math>\text{NH}_3</math>, confirms Br.</li> <li>Yellow ppt. insoluble in aq. <math>\text{NH}_3</math>, confirms I.</li> </ol>
Nitrogen and sulphur together	$\text{Na} + \text{C} + \text{N} + \text{S} \xrightarrow{\Delta} \text{NaCNS}$ <p>Sodium thiocyanate (Blood red colour)</p>	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 - Liebig'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

#### Dumas'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

**Priti Singhal**



## **Msc Organic Chemistry Practical Manual:**

*ORGANIC MEDICINAL CHEMISTRY PRACTICAL MANUAL FOR PHARMACY AND SCIENCE STUDENTS* Anees Ahmad Siddiqui, The manual illustrates the concept of basic techniques in practical organic medicinal chemistry It aims to meet the requirements of B Pharmacy students under the new syllabus prescribed by Pharmacy Council of India It will also be useful to BSc BSc Hons and MSc medicinal chemistry students

*A Systematic Course of Practical Organic Chemistry* Lionel Guy Radcliffe, 1905

*Classified Guide to Technical and Commercial Books*, 1904

*A Manual of Machine Drawing and Design* David Allan Low, Alfred William Bevis, 1898

*A Text-book of Physics* William Watson, 1904

*Laboratory Manual in Engineering Chemistry : For the Students of JNTU Hyderabad* Dr. B. Rama Devi & Dr. P. Aparna, The book is written to gain the basic knowledge on the principles of chemistry required for practical applications in engineering concepts This book consists organic and general chemistry experiments for chemical engineering for 1st and 2nd semester students The book also explains the precautions and safety rules for avoiding the accidents in chemistry laboratory It covers Estimation of Ferrous iron by Dichrometry and Permanganometry Method Estimation of Acetic Acid by Conductometric Titrations Estimation of the Amount of Fe<sup>2+</sup> by Potentiometry Determination of an Acid Concentration using pH Meter Preparation of Nylon 6 and Bakelite Phenol Formaldehyde Resin Estimation of Acid Value of Given Lubricant Oil Determination of Rate of Corrosion of Mild Steel Preparation of Benzanilide from Benzophenone via the Oxime by Beckmann Rearrangement etc

*Elementary treatise on physics, experimental and applied*, tr. and ed. by E. Atkinson Adolphe Ganot, 1898

*The Steam Turbine* Robert Morrison Neilson, 1902

**The Book of the Rifle** Thomas Francis Fremantle, 1901

*The Old Riddle and the Newest Answer* John Gerard, 1904

*Publisher and Bookseller*, 1905 Vols for 1871 76 1913 14 include an extra number The Christmas bookseller separately paged and not included in the consecutive numbering of the regular series

*The Bookseller*, 1905 Official organ of the book trade of the United Kingdom

**A Text-book on Ceramic Calculations** Willie Jackson, 1904

**The Organic Chem Lab Survival Manual** James W. Zubrick, 2016-01-19 Written for the laboratory that accompanies the sophomore junior level courses in Organic Chemistry Zubrick provides students with a valuable guide to the basic techniques of the Organic Chemistry lab The book will help students understand and practice good lab safety It will also help students become familiar with basic instrumentation techniques and apparatus and help them master the latest techniques such as interpretation of infrared spectroscopy The guide is mostly macroscale in its orientation

*Journal of the Society of Chemical Industry* Society of Chemical Industry (Great Britain), 1925 Includes list of members 1882 1902 proceedings of the annual meetings and various supplements

*Reference Catalogue of Current Literature*, 1913

*CBSE Class 12 Mastering Organic Reactions MCQ With Answers* Priti Singhal, 2024-11-11 The primary objective of this book is to serve as a comprehensive guide for students educators and researchers by focusing on reaction mechanisms practical applications and problem solving techniques Organic chemistry is not just about memorizing equations and formulas it is

about understanding how molecules interact change and influence each other under different conditions With that in mind this book emphasizes the logic and patterns behind organic reactions making it easier for readers to apply concepts across a variety of scenarios Each chapter of this book builds upon foundational knowledge ensuring a progressive learning experience From nucleophilic substitutions to pericyclic reactions and from oxidation reduction mechanisms to named reactions we cover both fundamental and advanced topics to cater to students at all levels Real world examples have been integrated throughout the chapters to show how organic reactions play essential roles in pharmaceuticals biochemistry agriculture and environmental science This approach bridges the gap between theory and practical applications helping readers appreciate the relevance of organic chemistry in daily life

**Natural Products** Raphael Ikan,2013-10-22 This new edition has been updated to include the following The use of biomarkers organic compounds in the geospherical record with carbon skeletons reflecting the upsurge in geoporphyrin research primarily due to MS yeast RNA nucleic acid studies reversed phase HPLC of amino acids brewing industry applications HPLC evaluation of carotenoids in orange juice and of debittered citrus HPTLC of carbohydrates synthesis of a sweetening agent from citrus peels synthesis and degradation of alkaloids and of sterols GC MS uses with sterols petroleum products and aromatic constituents of wine and grape juice flash chromatography of essential oils optical purity of enantiomers affecting flavors fragrances and pheromones as well as studies of lattice inclusion compounds <sup>1</sup>H and <sup>13</sup>C NMR MS IR and UV data are presented for most natural products Biomarkers organic compounds in the geospherical record with carbon skeletons reflecting the upsurge in geoporphyrin research primarily due to MS Yeast RNA nucleic acid studies Reversed phase HPLC of amino acids citrus juice components and HPLC in brewing industry application HPTLC of carbohydrates <sup>1</sup>H and <sup>13</sup>C NMR Sweetness evaluation and synthesis of a sweetening agent from citrus peels seed oil sesamol alkaloids strychnine piperine caffeine and sterol analyses GC MS sterols petroleum studies aromatic constituents of wine and grapejuice Flash chromatography of essential oils Optical purity of enantiomers affecting flavors fragrances and pheromones Materials science studies of lattice inclusion compounds

British Medical Journal ,1899      *Illustrated Catalogue of Chemical Apparatus, Assay Goods and Laboratory Supplies ...*  
Eimer & Amend,1910

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