

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	$\text{Na} + \text{C} + \text{N} \xrightarrow{\Delta} \text{NaCN}$	$(\text{NaCN} + \text{FeSO}_4 + \text{NaOH}) \xrightarrow{\text{boil and cool}} \text{FeCl}_3 + \text{conc. HCl} \rightarrow \text{Fe}_3(\text{Fe}(\text{CN})_6)_4$ 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) $\text{Na}_2\text{S} + \text{CH}_3\text{COOH} + (\text{CH}_3\text{COO})_2\text{Pb} \rightarrow$ 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

Anees Ahmad Siddiqui



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

Laboratory Manual of Organic Chemistry Raj K. Bansal, 2009 About the Book The manual has been thoroughly revised several new experiments and tests have been added while some redundant material has been deleted Chapter 2 has been completely rewritten An obvious change of this edition constitutes the splitting of Chapter 7 into two separate Chapters Tables on derivatives of organic compounds have been expended Also included are 20 estimations 75 preparations and isolation experiments and approximately 135 in text questions related to the experiments The approximation of modern spectroscopic techniques to structure determination have been discussed in the last Chapter This book is designed both for undergraduate and postgraduate level students with its enhanced and comprehensive presentation This is an indispensable book for organic chemistry practicals About the Author Dr Raj K Bansal received his M S from the University of California Davis Calif U S A and Ph D from Calgary University Calgary Alberta Canada He was a postdoctoral fellow at the National Research Council N R C of Canada in Halifax N S Canada followed by a Research Associateship at the Mellon Institute of Science Carnegie Mellon University Pittsburgh Pa U S A Dr Bansal has published a number of research papers in various foreign and Indian scientific journals He is the author of six books on chemistry including this work A Textbook of Organic Chemistry 5th ed 2007 Organic Chemistry Problems and Solutions 2nd edn 2006 and Heterocyclic Chemistry 4th edn 2005 One of his books Synthetic Approaches in Organic Chemistry has been reprinted by Jones and Bartlett Publishers Sudbury Massachusetts U S A Dr Bansal was a former Professor Department of Chemistry Indian Institute of Technology Delhi Hauz Khas New Delhi

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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
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 - Msc Organic Chemistry Practical Manual eBook Subscription Services
 - Msc Organic Chemistry Practical Manual Budget-Friendly Options
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 - 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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