FOR ALL PHARMACY STUDENTS

EXPERIMENTAL

PHARMACEUTICAL ANALYSIS-I

(VOLUMETRIC)

Dr. SANJAY G. WALODE

BHUSHAN M. FIRAKE

NIRALI PRAKASHAN

EXPERIMENTAL

PHARMACEUTICAL ANALYSIS - I

(VOLUMETRIC)

For All Pharmacy Students

Dr. Sanjay G. Walode
M. Pharm., Ph.D. (Pharmaceutical Chemistry)
Professor & HOD
STES's, Sinhgad Institute of Pharmaceutical Sciences
Kusgaon (Bk), Lonavala, Pune-410401, India

Prof. Bhushan M. Firake
M. Pharm. (Pharmaceutical Chemistry)
Assistant Professor
JSPM's Jaywantrao Sawant College of Pharmacy and Research
Hadapsar, Pune

Price ₹ 265.00

NIRALI PRAKASHAN
ADVANCEMENT OF KNOWLEDGE

N3932

Experimental Pharmaceutical Analysis - I (Volumetric) **ISBN 978-93-86943-15-6**

First Edition : August 2017

Published By :
NIRALI PRAKASHAN
Abhyudaya Pragati, 1312, Shivaji Nagar,
Off J.M. Road, PUNE – 411005
Tel - (020) 25512336/37/39, Fax - (020) 25511379
Email : niralipune@pragationline.com

Polyplate

Printed By :
YOGIRAJ PRINTERS AND BINDERS
Works: Sr. No. 10\1,Ghule Industrial Estate,
Nanded Village Road,
TAL-HAVELI, DIT-PUNE 411041.
Mobile - 9850046517, 9404225254

☞ DISTRIBUTION CENTRES

PUNE

Nirali Prakashan : 119, Budhwar Peth, Jogeshwari Mandir Lane, Pune 411002, Maharashtra
Tel : (020) 2445 2044, 66022708, Fax : (020) 2445 1538
Email : bookorder@pragationline.com, niralilocal@pragationline.com

Nirali Prakashan : S. No. 28/27, Dhyari, Near Pari Company, Pune 411041
Tel : (020) 24690204 Fax : (020) 24690316
Email : dhyari@pragationline.com, bookorder@pragationline.com

MUMBAI

Nirali Prakashan : 385, S.V.P. Road, Rasdhara Co-op. Hsg. Society Ltd.,
Girgaum, Mumbai 400004, Maharashtra
Tel : (022) 2385 6339 / 2386 9976, Fax : (022) 2386 9976
Email : niralimumbai@pragationline.com

☞ DISTRIBUTION BRANCHES

JALGAON

Nirali Prakashan : 34, V. V. Golani Market, Navi Peth, Jalgaon 425001,
Maharashtra, Tel : (0257) 222 0395, Mob : 94234 91860

KOLHAPUR

Nirali Prakashan : New Mahadvar Road, Kedar Plaza, 1st Floor Opp. IDBI Bank
Kolhapur 416 012, Maharashtra. Mob : 9850046155

NAGPUR

Pratibha Book Distributors : Above Maratha Mandir, Shop No. 3, First Floor,
Rani Jhanshi Square, Sitabuldi, Nagpur 440012, Maharashtra
Tel : (0712) 254 7129

DELHI

Nirali Prakashan : 4593/15, Basement, Aggarwal Lane, Ansari Road, Daryaganj
Near Times of India Building, New Delhi 110002
Mob : 09555778814

BENGALURU

Pragati Book House : House No. 1, Sanjeevappa Lane, Avenue Road Cross,
Opp. Rice Church, Bengaluru – 560002.
Tel : (080) 64513344, 64513355,Mob : 9880582331, 9845021552
Email:bharatsavla@yahoo.com

CHENNAI

Pragati Books : 9/1, Montieth Road, Behind Taas Mahal, Egmore,
Chennai 600008 Tamil Nadu, Tel : (044) 6518 3535,
Mob : 94440 01782 / 98450 21552 / 98805 82331,
Email : bharatsavla@yahoo.com

Acknowledgement

It is great pride and immense pleasure to extent my sincere regard and deepest sense of gratitude to Hon'ble Prof. M. N. Navale (Founder President), Hon'ble Dr. (Mrs.) Sunanda M. Navale (Founder Secretary), Sinhgad Technical Education Society, Pune for their continuous encouragement, constructive criticism and invaluable support for the publication of this book.

It is a pleasure moment for me to gratefully acknowledge the guidance and co-operation of Dr. R. N. Kane, Principal, Sinhgad Institute of Pharmaceutical Sciences, Lonavala, for his support, inspiration, help and guidance, which provided me, moral and confidence during the work.

I cordially acknowledge my gratitude to my beloved wife Trupti, my beloved daughter Anagha and my beloved son Aarush, who has always been a great source of inspiration and moral support throughout my writing endeavour.

The authors extend his due thanks to publishers Mr. Dineshbhai K. Furia and Mr. Jigneshbhai C. Furia of Nirali Prakashan, Pune, for their excellent support in the production of this experimental practical book in a record time period.

I take this opportunity to express my sincere thanks to Dr. S. B. Gokhale for his support, guidance and constant encouragement.

I express my heartfelt thanks to Mrs. Roshan Khan, Mrs. Manasi Pingle and other supporting staff members of Nirali Prakashan, for their helpful suggestions and skilful supervision which helps to mould the text in a beautiful book.

Last but not least, I would like to dedicate my book to my beloved parents, whose love and blessings save me an emotional support and the zeal to work hard towards my goal.

Dr. Sanjay G. Walode

Acknowledgement

I wish to express my profound gratitude and indebtness to Dr. K. G. Bothara for his indirect inspiration, endless motivation, and encouragements during this write-up.

I take this opportunity to thank to Dr. S. G. Walode, Dr. S. D. Firke, Dr. V. R. Patil, Dr. R. R. Somani, Dr. S. D. Ambavade, Dr. P. D. Chaudhari, Dr. V. I. Hukkeri for their valuable guidance for successful completion of this book.

I offer my sincere gratitude to Hon. Prof. T. J. Sawant, Dr. V. A. Bugade, and management of Jayawant Shikshan Prasarak Mandal (JSPM), Pune, Maharashtra for their indirect support and motivation.

Most importantly, I, Mr. B. M. Firake, would like to acknowledge my family members, Mr. Murlidhar M. Firake (my father), Mr. Kiran M. Firake (my brother), Mr. Milind J. Rane, Mr. Vilas S. Sarode and six women (My mother, Mrs. Kavita Murlidhar Firake, Vahini, Mrs. Hemangi Kiran Firake, mami, Mrs. Madhuri Vilas Sarode, mother-in-law, Mrs. Sunanda Jagannath Rane, daughter, Kavya Bhushan Firake, and my sweetest wife and life, Mrs. Tejal Bhushan Firake) for their generous help, criticism, motivation and for everything they have done in making me what I am today.

I am thankful to Mrs. Manasi Pingle, Mrs. Roshan Khan, Mr. Ravi Walodare and the staff members of Nirali Prakashan for their sincere efforts and co-operation and interest taken to shape this edition.

Prof. Bhushan M. Firake

Preface

Analytical chemistry is concerned with the chemical characterization of matter by both qualitative and quantitative ways. It gives us immense pleasure to introduce **"Experimental Pharmaceutical Analysis – I (Volumetric)"**. This book has been designed and arranged essentially to provide complete experimental knowledge of pharmaceutical analysis especially volumetric analysis to undergraduate students pursuing career in pharmaceutical sciences in India.

The contents of the book are structured so as to carry out volumetric analysis as per Pharmacy, undergraduate syllabus. Book is written in a simple and comprehensive manner along with the schematic diagrams and tables that clearly demonstrate core concept of volumetric analysis. The authentic text of the book will definitely furnish exhaustive information to the students with impressively and user-friendly style.

We will be grateful to all the students, teachers and readers for their constructive suggestions to improve the quality content of this book. The suggestions from all the readers will be highly appreciated and will be incorporated in the next edition.

Dr. Sanjay G. Walode

Prof. Bhushan M. Firake

Contents

Chapter … 1

INTRODUCTION TO ANALYTICAL CHEMISTRY

INTRODUCTION

Chemistry (generally called Central Science) deals with the study of matter, including its composition and structure, its physical properties, and its reactivity. Chemistry is measurement based science discipline which is generally based on number of other fields like organic chemistry, inorganic chemistry, biochemistry, physical chemistry, and analytical chemistry.

- **Organic chemistry** is the area of chemistry that involves the study of carbon and its compounds.
- **Inorganic chemistry** is the study of the synthesis, reactions, structures and properties of compounds of the elements.
- **Biochemistry** is said to be a hybrid science as it contain *Bio* (Biology- science of living organisms) and *chemistry* (science of atoms and molecules), so biochemistry is the science of the atoms and molecules in living organisms (such as DNA and proteins).
- **Physical chemistry** deals with the study of the physic chemical characteristics of the substance without changing its composition.

Analytical chemistry deals with methods for determining the chemical composition of sample. Analytical chemistry is found to be useful tool in nearly all aspects of chemistry viz agricultural, clinical, environmental, forensic, manufacturing, metallurgical, and pharmaceutical chemistry. The techniques of this science are used to identify the substances which may be present in a material and to determine the exact amounts of the identified substance.

Chemical analysis may be classified into four kinds:

(i) Proximate Analysis: It determines the amount of each element in a sample with no concern as to the actual compounds present.

(ii) Partial Analysis: It deals with the determination of designated constituents in the sample.

(iii) Trace Constituent Analysis: It is a specialized for the partial analysis determination of specified components present in very minute quantity.

(iv) Complete Analysis: It determines the proportion of each component of the sample.

Chemical analysis is often classified on the basis of sample size:

(i) Macro Analysis: This analysis deals with the quantities of 0.1 g or more.

(ii) Meso (semimicro) Analysis: This analysis deals with the quantities ranging from (0.01 g - 0.1 g).

(iii) Micro Analysis: This analysis deals with quantities in the range (0.001 g - 0.01 g).

(iv) Submicro Analysis: This analysis deals with samples in the range (0.0001 g - 0.001 g).

(v) Ultramicro Analysis: This analysis deals with quantities below 0.0001 g.

Major constituent: A major constituent is one accounting for 1 - 100%.

Minor constituent: A minor constituent exist in the range of 0.01 - 1%.

Trace constituent: A trace constituent is presented at an absorption of less than 0.01 %.

PHARMACEUTICAL ANALYSIS AND ITS SCOPE

Pharmaceutical analysis is a branch of chemistry, which deals with the process or sequences of processes to identify and/or quantify a substance or drug, the component of pharmaceutical solution or mixture, or the determination of the structure of chemical compound used in the formulation of pharmaceutical product.

Some specific uses of analysis are as follows:

(i) The examination of raw material, in-process and finished product.

(ii) Separation, determination and quantitation of impurity.

(iii) To check the strength and concentration of the chemical compound.

(iv) Used for identification and characterization of chemical compound.

(v) To determine the molecular weight of the chemical compound.

(vi) Used for structural elucidation of synthetic compound.

(vii) Quantitative analysis of air, water and soil samples is carried out to determine the level of pollution.

(viii) In farming, nature of soil and level of fertilizer application is analyzed.

(ix) In geology, composition of the rock and soil is carried out.

(I) DIFFERENT TECHNIQUES OF ANALYSIS

Analysis is broadly divided into two types:

- **Qualitative Analysis:** It gives information about the identity of atomic and molecular species or the functional groups in samples. It is used only to determine the presence and absence of the compound.
- **Quantitative Analysis:** It establishes the relative amount of one or more of the species (analyte) in numerical terms. It measures the concentration or amount of each substance in a sample.

Difference Methods of Analysis

1. Analytical Method:

Qualitative and/or quantitative analysis are carried out for one or more analytes using a specified technique/analytical method.

Classification of Analytical Methods:

Various methods of analysis can be broadly classified into two categories; Classical methods and Instrumental methods:

(i) Classical Methods:

These are based on traditional method of analysis and may be divided as:

Qualitative: Use for identification by colour, odour, boiling point, melting point, limit test etc.

Quantitative: Use for determination of mass or volume (e.g. volumetric, gravimetric).

- **Volumetric analysis** is used to determine the exact volume of the solution of known concentration required to react completely with the solution of the substance to be analysed.
  - **(i) Neutralization acid-base titration:** It involves neutralization acid base reaction in presence of water as solvent.
  - **(ii) Non-aqueous acid-base titration:** It involves reaction in between acid and base in presence of non-aqueous solvent i.e. organic solvent.
  - **(iii) Complexometric titration:** It is complex formation reaction. In this titration, the reacting substance reacts with standard solution to form a soluble but very slightly dissociated complex.
  - **(iv) Precipitation titration:** It is precipitation formation reaction. In this titration, the reacting substance reacts with standards solution to form a precipitate or slightly soluble salt.
  - **(v) Oxidation-reduction titration:** In this titration, simultaneous oxidation–reduction reaction occurs. It includes all the methods where one reacting substance is oxidized while other get reduced.
- **Gravimetric analysis** is quantitative determination of an analyte based on the mass of a solid by the process of isolating definite compound of element in pure form. In this technique, substance under determination is converted into an insoluble precipitate which is collected and weighed.

(ii) Instrumental Methods:

These methods are based upon the measurement of some physical properties as conductivity, electrode potential, light absorption or emission, mass-to-charge ratio and fluorescence of substance. When non-instrumental method is not possible, instrumental method is the only answer to the problem.

The instrumental methods have advantages of being less time consuming over classical methods. Various factors such as cost of instruments, sensitivity, accuracy, precision and reproducibility have to be taken into considerations before selecting method for analysis.

Qualitative: Chromatography, electrophoresis and identification by measuring physical property (e.g. spectroscopy, electrode potential) are qualitative methods.

Quantitative: These are used for measuring property and determining its relationship to concentration (e.g. spectrophotometry, mass spectrometry).

Often, same instrumental method is used for qualitative and quantitative analysis.

Advantages of Instrumental Methods:

1. A small amount of a sample is required for analysis.
2. Determination is comparatively fast.
3. Various complex mixtures can be analysed with or without their separation.
4. High sensitivity and selectivity.
5. Results obtained by instrumental method are reliable and accurate.

Limitations of Instrumental Methods:

1. Instrumental methods are comparatively costly.
2. Need of regular maintenance of instrument.
3. Skilled personnel required.
4. Specialized training is required for handling instrument.
5. The sensitivity and accuracy depends upon the type of instrument.
6. Selectivity of instrument is required for specific samples.
7. Some time cross checking of results with other methods is needed.

Types of Instrumental Methods:

There are many techniques available for the analysis of analytes, which can be broadly classified as:

(A) Electro analytical methods (Chemical potential + Electrons)

(B) Analytical separation methods (Chemical equilibrium + Detectors)

(C) Analytical spectroscopic methods (Chemical energy + Photons)

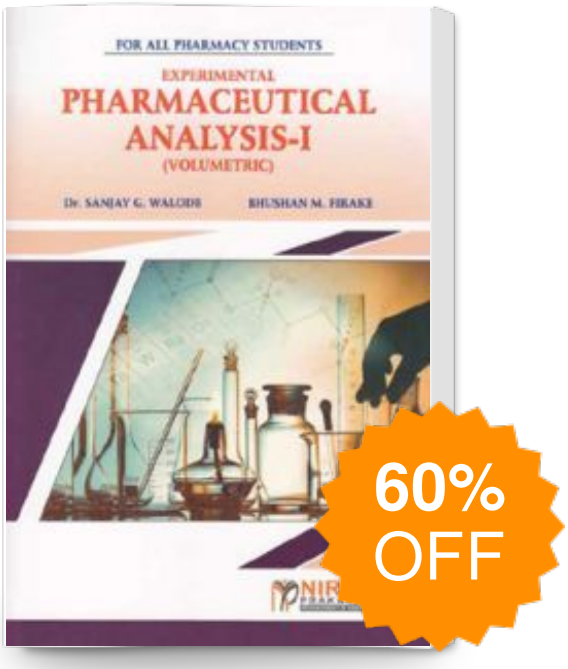
(D) Miscellaneous methods

(A) Electro Analytical Methods:

Electrochemical methods are powerful and versatile analytical techniques that offer high sensitivity, accuracy and precision with relatively low cost instrumentation.

1. **Potentiometry** is useful means of characterizing an acid, of which the potential is measured across the analyte without using indicator. It gives information about the composition of the sample through the potential appearing between two electrodes.
2. **Voltammetry** is the study of current as a function of applied potential. It is used to designate the current-voltage measurement obtained at a given electrode.

Experimental Pharmaceutical Analysis - I (Volumetric)



Publisher : Nirali Prakashan

ISBN : 9789386943156

Author : Dr. Sanjay G. Walode, Prof. Bhushan M. Firake