

FOR ALL PHARMACY STUDENTS

EXPERIMENTAL
PHARMACEUTICAL
ANALYSIS-I
(VOLUMETRIC)

Dr. SANJAY G. WALODE

BHUSHAN M. FIRAKE



 **NIRALI**
PRAKASHAN
ADVANCEMENT OF KNOWLEDGE

EXPERIMENTAL

PHARMACEUTICAL ANALYSIS - I

(VOLUMETRIC)

For All Pharmacy Students

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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

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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:

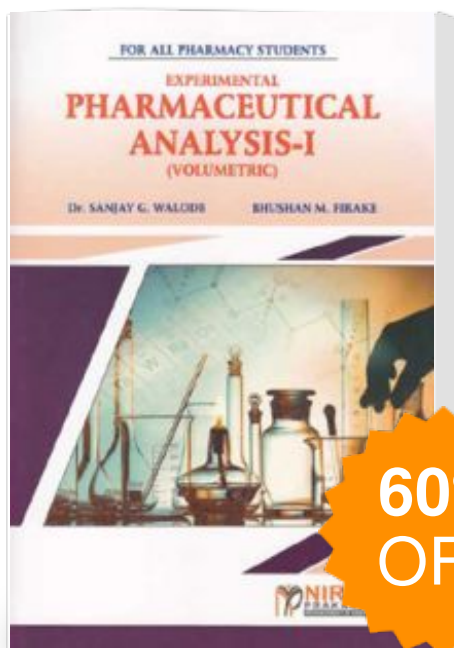
- (A) Electro analytical methods (Chemical potential + Electrons)
- (B) Analytical separation methods (Chemical equilibrium + Detectors)
- (C) Analytical spectroscopic methods (Chemical energy + Photons)
- (D) Miscellaneous methods

(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.

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