PHARMACEUTICAL ANALYSIS

INTRODUCTION TO PHARMACUTICAL ANALYSIS

⇒ THE Pharmaceutical Analysis is a Branch of Chemistry, Which involves the series of process for the -

- IDENTIFICATION
- DETERMINATION
- QUANTITATION
- PURIFICATION

⇒ This is mainly used for the separation of the components from the mixture and for the determination of the structure of the compounds.

⇒ The different Pharmaceutical agents as- follow -

[A] PLANTS.
[B] MINERALS.
[C] MICROORGANISMS
[D] Synthic compounds.

⇒ ANALYTICAL CHEMISTRY IS DIVIDED INTO — TWO- PARTS.

[A] [QUALITATIVE ANALYSIS] — This Method is used for the Identification of elements, IONS, and Chemical Compounds.

[B] - [Quantitative Analysis] — This Method is used for the Determination of the Individual elements, or Compound Present in a Sample, or substance

⇒ PHARMACEUTICAL - USED IN Mainly ↓

METHODS OF QUANTITATIVE ANALYSIS

⇓

This Method is used for the Determination of the Amount of the sample

→ There are various Methods OF Quantitative analysis

→ They are classified into the Following Types

① CHEMICAL METHODS.

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[A] Volumetric or, titrimetric

[B] GRAVIMETRIC

[C] GASOMETRIC.

[II] INSTRUMENTAL, or, PHYSICO-CHEMICAL Methods.

[III] MICROBIOLOGICAL Methods.

[IV] BIOLOGICAL Methods.

* Volumetric Methods:

In Volumetric or, titrimetric Methods, a solution of one reactant of accurately known concencentration (Standard Solution) is added to a second reactant (the Solution of whose concentration or amount to be determined).

* Volumetric Method is classified into different types depending upon the types of Reaction.

[A] Acid-Base, or Neutralisation titration.

Ex-> Hydrochloric Acid [HCl]
-> Sodium Hydroxide [NaOH

[B] Non-Aqueous titration.

Ex-> Perchloric Acid
-> Sodium Methoxide.

[C] Oxidation-Reduction titration.

Ex-> Oxidizing Agent: -> Iodine
-> Potassium Permanganate.
-> Potassium dichromate.

Ex-> Reducing Agent.
Ex -> Sodium thiosulphate
-> Oxalic Acid
-> Ammonium Iron(II) Sulphate [Mohrs salts]

[D] Precipitation Titration: - Ex-> AgCl

[E] Complexometric titration: - Ex-> EDTA [ethylenediaminetetraacetic acid]

* Gravimetric Analysis ->

Analysis By Weight is the process of Isolation and weighing an element or a definite compound of the element in a pure form as possible.

* Gasometric Methods: - The gasometric Methods Involve mesurment of the volume of gases. Ex - Nitrous oxid, Carbon dioxide.

(II) Instrumental Methods ⇒

[A] Electrochemical Methods ⇒

(i) → Conductometry → mesurment of Conductance.

(ii) → Potentiometry → Mesurment of Potential.

[iii] → Coulometry → mesurment of current.

[iv] Voltametry → mesurment of specific valley.

[B] → Optical Methods ⇒

Based upon the Mesurment of Radiation - absorbed or emitted.

Ex → * Absorbation Methods →

- I.R [Infrared]
- U.V - [Ultraviolet]
- N.M.R

* → Emission Methods :

- Flame spectroscopy.
- Fluorimetry.

[C] Thermal Method →

Ex → Differential Thermal Analysis [DTA]

→ Differential Scanning Calorimetry [DSC]

[D] - Chromatography → Ex → HPLC
→ TLC
→ HPTLC
→ GC
→ Column Chromatography
→ Ions - Exchange

* → Supercritical fluid Chromatography

* → Affinity Chromatography

[E]. OTHER Methods →

Ex → X-Ray. diffractometry.

→ Mass ~~spectroscopy~~.
Spectrometry.

③ MICROBIOLOGICAL Methods.

⇓

THE INHIBION OF MICROBIAL GROWTH UNDER the STANDARDIZED CONDITIONS May BE UTILISED FOR KNOWING the THERAPEUTIC EFFICACY OF ANTIBIOTICS.

⟹ They are based upon a Comparison of the Inhibition of growth of bacteria By Measured of /Antibiotic ~~having~~ ~~known~~ - ~~Activity~~. to examined with Produced By known concentration of Standard Preparation of the antibiotic having know activity.

* ⟹ Two - generally Methods – [A] cup-Plate methods.
[B] cylinder Plate methods.

[4] Biological Method ⟹ Biological Standardisation, or, Bioassays are the Procedure By which the Potency or the Nature of the Substance is estimating By studing its effects of living matter.

⟹ Bioassays are generally done using animal tissue or organ as the case of use of guinea Pig Ileum for the estimation of HISTAMINE.

⟹ Bioassays are designed to measure the Relative Potency of two Preparation - usually a standard and unknown.

✳ FUNDAMENTAL CONCEPTS IN VOLUMETRIC ANALYSIS.

✳ Solute → THE substance WHICH DISSOLVED IN the solvent.

✳ Solvent → THE substance WHICH is dissolving the solute. or,

✳ Solution → It is a Homogenous mixture of two or more components.

✳ Saturated solution → Both the concentration of Solute and Solvent are at equilibrium.

✳ Supersaturated Solution → Solute concentration is High.

✳ Unsaturated Solution → Solute concentration is low.

✳ VERY Soluble → Less than ONE PARTS of solvent Required.

✳ Freely Soluble → 1 to 10 PARTS.

* Soluble → 10 to 30 PARTS

* Strongly Soluble → 30 to 100 PARTS.

* Slightly Soluble → 100 to 1000 PARTS.

* VERY Slightly Soluble → 1000 to 10000 PARTS.

* Practicaly INsolube → More than 100000 Parts.

⇒ Based on the Solute dissolved In the Solvent Again Solutions.

- Gases In liquids — Ex Ammonia dissolved In Water.
- liquids IN liquids → Ex - Alcohol- dissolved IN Water.
- Solids IN liquids → Ex- KOH dissolved In water.

* CO-SOLVENCY :→ The solubility of the Poorly Soluble Compound is enhanced By the Addition OF Co-Solvents.

→ Co-Solvents are the mixture of Water and water miscible Solvents.

Ex ⇒ PEG-300
⇒ Ethanol
⇒ Propylene glycol.

* PPm and PPb → PARTS PER Million and PARTS PER BILLION (PPb)

$$\text{CONCENTRATION OF PPm} = \frac{\text{Mass of solute}}{\text{mass of solvent}} \times 10^6 \text{ PPm}$$

* PRIMARY STANDARD :

- It should Be stable.
- It should Be completely soluble IN solvent.
- Its Purity can be determined By standard analytical methods
- The reaction With the standard solution Should Be Stoichiometric reaction.

Ex⇒ EDTA, SUCCINIC Acid, Sodiuma Carbonate

* ⇒ SECONDARY STANDARD ⇒

The substance which is not Primary standard in Nature is called a secondary standard.

- Scondary standard should be stable long Period of time
- It should will Rabidaly Reacts.
- It should Prodade SHARP end Points.

STANDARD Solution ⇒ The solution of Accurately known strength is called the Standard solution

EQUIVALENCE POINT ⇒ The Point in a titration where the reaction is just completed.

* INDICATOR ⇒ These are very Weak organic Acid or, Bases. Based on Hydrogen Ion concentration present In the solution the indicator, produce different Colours.

⇒ Titration to located the end-Point is called a titration.

(A) ⇒ ONE-COLOUR INDICATOR ⇒ Ex- Phenolphthalein which Produce- Pink colour

[II] Two-colour Indicator → Ex- Methyal orange to Produce Red to yellow colour

[III] Mixed-Indicator → Ex= Natural Red and methylene Blue

Example:-

	INDICATORS	COLOUR IN ACID		COLOUR IN BASE
[1]	Methyl orange →	Red	→	Orange
[2]	Methyl-yellow →	Red	→	yellow
[3]	Methyl-Red →	Red	→	yellow
[4]	Phenolphthalein →	Colorless	→	Red
[5]	Phenol-Red →	yellow	→	Red
[6]	Thymole Blue →	Red	→	yellow
[7]	Bromo Phenol Blue →	yellow	→	Blue
[9]	Bromo Cresol green →	yellow	→	Blue

Titrant → This is a solution of the known concentration of the standard substance, which is added to the sample solution from the Burret.

Titrand: → This is a solution of the unknown sample whose concentration is to be determined.

Equivalence Point → This point where the reaction Between the titrant and titrand are Completly-and it can be detected By colour Change of the indicator.

✳ FORMALITY ⟹ (F)

The Number of Mole of Solute Per Litre of Solution with regardless to the Chemical Formula of the Compound.

✳ Molarity :⟹ (M) The Number of Mole of Solute Per Litre of Solution with regardless to Chemical Formula of the compound.

✳ Molality :⟹ (m) The Number of mole of the Solute in 1 kg of the Solution.

✳ Normality :⟹ (N) ⟹ The Number of ~~mole~~ equivalent of the Solute IN 1 Litre of the Solution.

✳ Equivalent wt ⟹ The Weight of the one equivalent Compound.