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PHARMACEUTICAL ANALYSIS I

UNIT 1

TOPIC :

- **Pharmaceutical analysis-** Definition and scope

  i) Different techniques of analysis

  ii) Methods of expressing concentration

  iii) Primary and secondary standards.

  iv) Preparation and standardization of various molar and normal solutions

  Oxalic acid, sodium hydroxide, hydrochloric acid, sodium thiosulphate,

  sulphuric acid, potassium permanganate and ceric ammonium sulphate

Pharmaceutical analysis

- Pharmaceutical analysis is branch of Pharmaceutical chemistry that involves a series of process for identification, determination, quantification and the purification of a substance, Seperation of the components of solution or mixture or the determination of structure of chemical compounds
- The substance may be single compound or a mixture of compounds and it may be in any of the dosage form. The substance used as pharmaceuticals are animals, plants, micro arganisms, minerals & various synthetic products.

Scope of Pharmaceutical Analysis

- Examination of raw material.
- Analysis of various drugs samples.
- qualitative /Quantitative analysis of samples.
- Diagnosis of various disease by Chemical analysis.
- Determination of radioactive compounds.
- Determination of different samples of water.

Techniques of analysis

a) Chemical method : In chemical method, for analysis of any element or compound we use different types of chemicals for testing its limits, organoleptic properties, physical properties and biological properties.

Different techniques used in chemical method are below

- Volumetric or titrimetric
- Gravimetric
- Gasometric

b) Instrumental methods : In instrumental method for analysis of any compound we use different types of laboratory instruments or glasswares. Some

- Spectrophotometry
- Fluorimetry
- Potentiometry
- Polarography
- Polarimetry,

C) Microbiological method : In this method, we use different types of microbes such as bacteria, virus fungi, algae.

Two general methods are usely employed -

- Cylindrical plate method
- Tube assay method

d) Biological method : In this method of bio assays the procedure by which potency or the nature of the substance is estimated by studying its effect on living matter.

Methods of expressing concentration

- These are those methods which are used to find out the concentration/amount of drug present in any solution.
- Concentration, is basically the amount of solute mixed with solvent.
- They are expressed by following terms :-

1) Molarity :

→ It is defined as the number of moles of the solute dissolved in one litre of solution

→ It is denoted by 'M'

$$\text{Molarity (M)} = \frac{\text{Moles of solute}}{\text{Volume of solution (in litre)}}$$

2) Normality :

→ It is defined as the number of gram equivalents of the solute dissolved per litre of solution.

→ It is denoted by 'N'

$$\text{Normality (N)} = \frac{\text{gram equivalents of the solute}}{\text{Volume of solution (in litre)}}$$

3. Molality :

→ It is defined as the number of moles of the solute per kilogram (kg) of the solvent.

→ It is denoted by 'm'

$$\text{Molality (m)} = \frac{\text{moles of solute}}{\text{Mass of solvent (in kg)}}$$

4) Formality :

→ It is defined as the number of gram formular weight of a solute dissolved in one litre of Solution.

→ It is denoted by 'F'

$$\text{Formality (F)} = \frac{\text{gram formular weight (GFW)}}{\text{Volume of solution (in litre)}}$$

5) Mole fraction :

→ It is define as, the ratio of no. of moles of solute to the total no. of moles of solute and solvent.

$$\text{Xsolute} = \frac{\text{moles of solute}}{\text{moles of solute + moles of solvent}}$$

$$\text{Mole percentage} = \text{Mole fraction} \times 100$$

6) Percentage Calculation :

→ Also known 'percentage concentration".

a) % by weight of solute

$$\%\ w/w = \frac{\text{wt. of solute}}{\text{Wt. of solution}} \times 100$$

b) % by volume of solute

$$\%\ v/v = \frac{\text{volume of solute}}{\text{Volume of solution.}} \times 100$$

c) % of weight of solute by vol. of solution.

$$\% \text{ w/v} = \frac{\text{weight of solute x 100}}{\text{Volume of solution}}$$

7) Parts per million (ppm) :

→It is the parts of solute in one million parts of solution.

$$\text{PPM} = \frac{\text{mass of solute}}{\text{mass of solution}} \times 10^6$$

STANDARD SOLUTIONS

- These are those solutions which have accurately known concentration and which is highly pure and which further use for standardization
- Standardization - to make solution standards.
- It is of two types :
    - Primary standards
    - Secondary standards

i) Primary Standards

These are those solution which are prepared through highly pure reagents or chemicals and they have accurately known concentration. they do not required further standardization.

Properties :

- Highly pure, less reactive and stable.
- Highly soluble, non-toxic and eco-friendly.
- **Example :** Sodium Carbonate, oxalic acid, silver nitrate etc

ii) Secondary standards

These are those solution which are less stable and standardized by using primary standard solutions. They are mainly used for quantitative analysis. They are used for standardization of other substances.

Properties :

- less pure and more reactive than Primary Standards
- less stable
- **Example :** Sulphuric acid (H_2SO_4), Potassium permanganate ($KMnO_4$), HCI (Hydrochloric acid) etc

Preparation and standardization of various molar and normal solutions

Oxalic acid solution

Preparation

- Weigh 6.3 gm of oxalic acid & dissolve in distilled water &finally make up the volume to one liter in a volumetric flask.
- The standard solution of oxalic acid (Primary standard) is used to find the strength of solutions of alkalies like NaOH, KOH (Secondary standards) whose standard solutions can not be made by direct weighing.

SINCE IT IS A PRIMARY STANDARD THERE IS NO NEED TO STANDARDIZE IT

SODIUM HYDROXIDE

Preparation

- 1 Normal solution we need to know the, equivalent of NaOH, which is calculated by dividing Molecular weight by 1, that is 40 divided by 1= 40. So the equivalent weight of NaOH is 40.
- To make 1 N solution, dissolve 40.00 g of sodium hydroxide in water to make volume 1 liter.
- For a 0.1 N solution (used for wine analysis) 4.00 g of NaOH per liter is needed.

Method of standardization

- Weigh accurately about 2.0 g of potassium hydrogen phthalate, previously powdered and dried at 120° for 2 hours, and dissolve in 75 ml of carbon dioxide-free distilled water.
- Add 0.1 ml of phenolphthalein solution and titrate with the sodium hydroxide solution until a permanent pink colour is produced.
- Each ml of IM sodium hydroxide is equivalent to 0.2042 g of $C_8H_5KO_4$

HYDROCHLORIC ACID

Preparation

- Dilute 85.0 ml of concentrate hydrochloric acid with purified water to produce 1000 ml

Method of standardization

- Weigh accurately about 1.5 g of anhydrous sodium carbonate, previously heated at about 270° for 1 hour. Dissolve it in 100 ml of distilled water and add 0.1 ml of methyl red solution
- Add the acid slowly from a burette, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool and continue the titration.
- Heat again to boiling and titrate further as necessary until the faint pink colour is no longer affected by continued boiling.
- Each ml of 1 M hydrochloric acid is equivalent to 0.05299 g of Na_2CO,

SODIUM THIOSULPHATE

Preparation

- Dissolve 25 g of sodium thiosulphate and 0.2 g of sodium carbonate in carbon dioxide-free distilled water and dilute to 1000 ml with the distilled water

Method of standardization

- Take 10 ml of Potassium Iodate solution Add 2 gm of Potassium Iodide and 5 ml of dilute H_2SO_4, keep it in dark for 10 minutes, add 2 to 3 drops of starch indicator and titrate with sodium thiosulphte using starch solution as indicator until the blue colour is disappeared.

$$N_1V_1 = N_2V_2$$

$$N_2 = N_1V_1 V_2$$

SULPHURIC ACID (H_2SO_4)

Preparation

- Take 54 ml of conc. H_2SO_4 in about 600 ml of H_2O in 1000 ml of volumetric flask; cool & finally make volume to 1000 ml with distilled water.

Method of standardization

- Take about 3.0 gm of anhydrous Na_2CO_3 dried previously at 270* C for one hour.
- Dissolve it in 100 ml of distilled water, add 0.1 ml of methyl red as indicator & titrate with 1 M H_2SO_4 with constant stirring until the solution becomes faintly pink.

- Heat the solution to boiling cool & continue the titration until the faint pink colour is no longer affected by continue boiling.
- Each ml of 1 M H_2SO_4 is equivalent to 0.10598 gm of Na_2CO_3 (Sodium Carbonate).

CERIC AMMONIUM SULPHATE

Preparation

- Dissolve 65 g of ceric ammonium sulphate, with the aid of gentle heat, in a mixture of 30.0 ml of sulphuric acid and 500 ml of distilled water.
- Cool, filter the solution, if turbid, and dilute to 1000 ml with distilled water.

Method of standardization

- Weigh accurately about 0.2 g of arsenic trioxide, previously dried at 105° for 1 hour, and transferred to a 500-ml conical flask.
- Wash down the inner walls of the flask with 25 ml of a 8.0% w/v solution of sodium hydroxide, swirl to dissolve, add 100 ml of distilled water and mix. Add 30 ml of dilute sulphuric acid, 0.15 ml of osmic acid solution, 0.1 ml of ferroin sulphate solution and slowly titrate with the ceric ammonium sulphate solution until the pink colour is changed to a very pale blue, adding the titrant slowly towards the end- point.
- Each ml of 0.1 M ceric ammonium sulphate is equivalent to 0.004946 g of As_2O_3

POTASSIUM PERMANGANATE

Preparation

- Dissolve 3.2g of potassium permanganate in 1000ml of water, heat on a water bath for 1 hour, allow to stand for 2 days. Filter the solution through glass wool.

Standardisation

- Take 20 ml of Oxalic acid solution add 5 ml of im sulphuric acid. Warm the mixture to about 700C titrate with potassium permanganate solution taken in the burette. End point is appearance of pink colour that persist for 30sec.
    - $N_1V_1 = N_2 V_2$
    - $N_2 = N_1V_1/V_2$