Analytical Chemistry

INTRODUCTION

Sawa University

College of Pharmacology

First Stage

جامعـــــــــة ســــــــاوة الأهلية

كلية الصيدلة

المرحلـــــــــــــــة الاولى

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المحاضرة الأولى : الجانب النظري

(a) PHARMACEUTICAL ANALYSIS:--

DEFINITION:--

Pharmaceutical analysis is branch of chemistry which is used to **identify**, **separate** and **purify** the substances.

It is used to determine ingredients, presence of impurities and excipients in pharmaceutical products and also used to determine uniformity, solubility and dissolution rate for such substances which are generally obtained from animals, plants, microbes, minerals **and** synthetic chemicals.

Pharmaceutical analysis **focuses on accessing the quantities and qualities of drugs and their impurities in development processes. It is also used for the separation of components from the mixture and for the determination of the structure of the compounds**.

SCOPE OR APPLICATIONOF PHARMACEUTICAL ANALYSIS:--

(1) Pharmaceuticalanalysis is basically used for the purpose of classification of compound according to its chemical properties.

(2) It is used for analysis of mixtures of compounds.

(3) It is used for the purpose of separation of components from mixture.

(4) It is used for purpose of purification, identification and characterization of compounds.

(5) It is also used to determine the quality and quantity of raw materials and finished products or formulations by using different methods of analysis.

(i) DIFFERENT TECHNIQUES OF ANALYSIS:--

It can be divided into two major categories:--

(1) QUALITATIVE ANALYSIS:--

(2) QUANTITATIVE ANALYSIS:--

(1) QUALITATIVE ANALYSIS:--

It is such type of analysis which is used to determine the composition of natural and synthetic products. It is used to indicate whether the substance or compound is present in the sample or not. It is also used for detection of evolved gas, for formation of precipitate,limit tests, colour change reactions, melting point and boiling point.

(2) QUANTITATIVE ANALYSIS:--

It is used to quantify any compound or substance in the sample by the use of different methods like

(a) gravimetric analysis.

(b) appropriate electrical measurements, for example, Potentiometry.

(c) the measurement of certain optical properties, for example; absorption spectra.

(d) in some cases like a combination of optical or electrical measurement and quantitative chemical reaction, for example, amperometric titration.

VARIOUS TYPES OF QUALITATIVE ANALYSIS:--

(1) Chemical method

(a) Volumetric or titrimetric method

(b) Gravimetric method

(3) Instrumental method

(4) Microbiological method

(5) Biological method

(1) CHEMICAL METHOD:--

(a) VOLUMETRIC METHOD:--

It is such type of method which is used to find out the volume or strength or normality or weight of unknown substance or solution by the help of strength of known substance or solution by finding out the volume of consumed solution by means of titration in the presence of suitable medium and indicator.

Further by applying certain

$$N_1V_1 = N_2V_2$$

formula, the normality or strength can be determined, where N_1, N_2 are normality of unknown and known solution and

V_1,V_2 are taken volume and consumed volume of the solution.

Volumetric analysis can be subdivided into following methods or titrations:-
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(1) Neutralization titration

(2) Non aqueous titration

(3) Redox titration

(4) Precipitation titration

(5) Complexometric titration

(b) GRAVIMETRIC METHOD:--

It is such type of method which is used to find out the weight of compounds by isolating and weighing of compounds by means of precipitation or volatilization method of analysis and converting them into pure form of compound.

Further with the help of known weights of constituents, presence of unknown substance can be calculated.

(2) INSTRUMENTAL METHOD:-- It is such type of method which is used to analyze the physical or chemical property of compound by the use of known strength of compound.

(3) MICROBIOLOGICAL METHOD:--

It is used for such type of compounds which are used for determination of inhibition of growth of bacteria by the substance to be analyzed in comparison with the standard compounds on the basis of result in respect of therapeutic efficacy of antibiotics.

(4) BIOLOGICAL METHOD:--

It is such type of method which is used to observe the biological effect of drug on some type of living matter. It is used to estimate the potency of drug.

It is also used to measure various parameters including weight of tissue of organ, weight of organs of blood, parameters such as blood, glucose, cholesterol, urea, enzyme etc.,

(ii) METHODS OF EXPRESSING CONCENTRATION:--

The concentration of solutions can be expressed quantitatively by different methods:--

(I) MASS PERCENTAGE (w/w):--

The mass percentage of a component can be defined as

$$\text{Mass\% of a component} = \frac{\textit{Mass of the component in the solution}}{\textit{Total mass of the solution}} \times 100$$

For example,

If a solution is described by 10% glucose in water by mass, it means that 10g of glucose is dissolved in 90g of water resulting in a 100g solution.

Concentration described by mass percentage is commonly used in industrial chemical applications.

For example, commercial bleaching solution contains 3.62 mass percentage of sodium hypochlorite in water.

(II) VOLUME PERCENTAGE:--

The volume percentage can be defined as

$$\text{Volume \% of a component} = \frac{\text{Volume of the component}}{\text{Total volume of solution}} \times 100$$

For example,

10% ethanol solution in water means that 10ml. of ethanol is diluted to total volume of 100 ml by using 90 ml of water.

Solutions containing liquids are commonly expressed in this unit.

For example, a 35%(v/v) solution of ethylene glycol is used in cars for cooling the engine.

(III) MASS BY VOLUME PERCENTAGE(w/v):--

Mass by valume percentage is commonly used in medicine and pharmacy. It is the mass of

the solute dissolved in 100 ml. of the solution.

$$W/V\% = m(g)/VmL$$

(IV) PARTS PER MILLION:--

When a solute is present in trace quantities then it is convenient to express concentration in parts per million (ppm).

Parts per million can be defined as

$$\text{Parts per million} = \frac{\text{Number of parts of the component}}{\text{Total number of parts of all components of the solution}} \times 10^6$$

$$ppm = m(\mu m)/VmL$$

Note:--

As in the case of percentage, concentration in parts per million can also be expressed as mass to mass, volume to volume and mass to volume.

A litre of sea water (which weighs 1030g) contains about 6×10^{-3} g of dissolved oxygen.

Such a small concentration is also expressed as 5.8g per 10^6g (5.8ppm) of sea water.

The concentration of pollutants in water or atmosphere is often expressed in terms of $\mu g\ mL^{-1}$ or ppm.

(V) MOLE FRACTION:--

Mole fraction of a component can be defined as

$$\text{Mole fraction of a component} = \frac{\text{Number of moles of the component}}{\text{Total number of moles of all the components}}$$

Mole fraction is commonly used by the use of symbol x and subscript used on the right hand side of x denotes the component.

For example,

In a binary mixture, if the number of moles of A and B are nA and nB respectively, the mole fraction of A will be

$$xA = \frac{nA}{nA + nB}$$

For a solution containing i number of components,

we have

$$x_i = \frac{n_1}{n_1 + n_2 + n_3 - - - - - - + n_i} = \frac{n_1}{\sum n_i}$$

It can be shown that in a given solution sum of all the mole fractions is unity i.e.,

$$x_1 + x_2 + - - - - - - - - - + x_i = 1$$

Mole fraction unit is very useful in relating some physical properties of solution, say vapour pressure with the concentration of the solution and quite useful in describing the calculations involving gas mixtures.

(VI) MOLARITY:--

Molarity(M) is defined as number of moles of solute dissolved in one litre (or one cubic decimeter) of solution.

$$Molarity = \frac{Moles\ of\ solute}{Volume\ of\ solution\ in\ litre}$$

For example,

0.25MolL^{-1} (or 0.25M) solution of NaOH means that 0.25mol of NaOH has been dissolved in one litre (or one cubic decimeter) water.

(VII) MOLALITY:--

Molality (m) is defined as the number of moles of the solute per kilogram (Kg) of the solvent.

It can be expressed as

$$Molality\ (m) = \frac{Moles\ of\ solute}{Mass\ of\ solvent\ in\ Kg}$$

For example, 1.00 mol Kg^{-1} (or 1.00m) solution of KCl means that 1 mol (74.5g) of KCl is dissolved in Kg of water.

Each method of expressing concentration of the solutions has its own merits and demerits. Mass percentage, mole fraction and molality are independent of temperature whereas molarity is a function of temperature. This is because volume depends on temperature and the mass does not.

(iii) PRIMARY AND SECONDARY STANDARDS:--

PRIMARY STANDARDS:--

Primary standards are such type of substances which are available in a pure state and their solution of definite normality can be prepared by weighing out an equivalent or a definite fraction by dissolving it in the solvent usually water and making up the solution to a known volume.

In practice a little more concentrated solution is prepared and then it is diluted with distilled water until the desired normality is obtained.

For example,

If N_1 = required normality, V_1 = volume after dilution,

N_2 = the normality originally obtained,

and V_2 = the original volume taken. then by applying following

formula,

V_1 or volume after dilution can be calculated. $N_1V_1 = N_2V_2$

Or,

$$V_1 = \frac{N_2 V_2}{N_1}$$

Examples of primary standards in different reactions can be listed as such:--

(i) ACID BASE REACTIONS:--

Sodium carbonate, sodium tetraborate, potassium hydrogen phthalate, constant-boiling point hydrochloric acid, potassium hydrogen iodate, benzoic acid.

(ii) COMPLEX FORMATION REACTIONS:--

Silver, silver nitrate, sodium chloride, various metals like zinc, magnesium, copper and spectrocopically pure manganese and salts depending upon the reaction used.

(iii) PRECIPITATION REACTIONS:--

Silver, silver nitrate, sodium chloride, potassium chloride and potassium bromide.

(iv) REDOX REACTIONS:--

Potassium dichromate, potassium bromate, potassium iodate, potassium hydrogen iodate, iodine, sodium oxalate, arsenic (III) oxide and pure iron.

SECONDARY STANDARDS:--

Seconday standards are such type of substances which are not available in pure state and their normality can be obtained approximately and definite normality can be calculated by means of standardization with primary standard substances.

Examples:--

(1) ACID BASE REACTIONS:--
Sodium hydroxide, potassium hydroxide, hydrochloric acid.

(ii) REDOX REACTIONS:--

Potassium permanganate, sodium thiosulphate, hydrogen peroxide.

(iii) COMPLEX FORMATION REACTION:--

Disodium EDTA

(iv) PRECIPITATION REACTIONS:--

Ammonium thiocyanate.

REUISITIES OF PRIMARY STANDARD:--

(1) **Primary standard must be easy to obtain, to purify, to dry** (preferably at 110 - 120 C) and to preserve in a pure state.

(2) The substance should be **unaltered in air during weighing**. Hence, it should not be hygroscopic nor oxidized by air nor affected by carbon dioxide. It should maintain its composition and should be unchanged during storage.

(3) The substance should be **capable of being tested for impurities** by qualitative and other tests of known sensitivity. The total amount of impurities should not exceed 0.01-0.02 percent.

(4) It should have a **high equivalent** so that the weighing errors may be negligible.

(5) The substance should be **readily soluble under the conditions** in which it is employed.

(6) **The reaction with the standard solution should be stoichiometric and practically instantancous**. The titration error should be negligible or easy to determine accurately by experiment.