DEPTH OF BIOLOGY

UNIT-I **10 Hours**

(a) 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

(b)Errors: Sources of errors, types of errors, methods of minimizing errors, accuracy, precision and significant figures

(c)Pharmacopoeia, Sources of impurities in medicinal agents,limit tests.

DEPTH OF BIOLOGY

Pharmaceutical Analysis

Definition :- It is the branch of pharmaceutical chemistry which involves the process of identification, determination, quantification and purification of substances.

- Application of analytical procedure which are used to determine the quality, safety and efficacy of drug and chemical.
- Purity must be check at every step.

DEPTH OF BIOLOGY

Importance :-

1. To identify the drug in formulated product.
2. Determination of Impurities or active ingredient to determine the stability of drug rate of release of a drug from its formation.
3. To identify the purity of drug.
4. To determine the concentration of specified impurities.
5. To determine the concentration of drug in plasma or in biological fluids.
6. To determine the parturient coefficient and solubility of drugs.

DEPTH OF BIOLOGY

Qualitative

To identify the constituent in mixture.

- Major constituent → >=1%
- Minor constituent → .01 – 1 %
- Trace constituent → .01 %

Quantitative

To identify the concentration or to the determine the concentration of different constituent in mixture.

DEPTH OF BIOLOGY

Various type of Analysis:-

1. *Chemical Analysis*

a) Volumetric analysis/ Titametric analysis

b) Gravimetric analysis

c) Gasometric analysis

2. *Electrical method*

a) Conductometry

b) Potentiometry

c) Polarography

DEPTH OF BIOLOGY

3. Instrumental Method

a) Spectroscopic analysis

b) Emission spectroscope /method

c) Mass spectroscope

d) Chromatography technique

4. Biomagical and Bio microbiological Method

a) Bioassay

b) Microbiological Methods

Molarity[M] → No. of moles of solute present in 1000ml of solution.

$$\text{No. of moles} = \frac{\text{wt. in gm}}{\text{mol. Wt.}}$$

Normality [N] → No. of equivalent of solute contain in 1L of solution.

$$\text{Equivalent wt.} = \frac{\text{molecular weight}}{\text{Valency or No. of H exchange}}$$

DEPTH OF BIOLOGY

Primary Standard :- It is a ultrapure compound that serve as the reference material for a titration.

Example → For strong acid solution, we use pure basic compound like Na Carbonate, Na Borate etc.

Properties of primary standard:-

- It should be 100% pure, impurity in range of 0.01%-0.002% is acceptable.
- It should be thermostable
- Eco friendly
- Non toxic

DEPTH OF BIOLOGY

- Less reactive
- Less hygroscopic and efflorescence
- It should not be expensive
- It should be readily available
- It should be dry before use
- It must have all the properties require for titration.

Secondary Standard :- whose purity has been determined by chemical analysis.

Another standard solution which are exclusively use for standardization of unknown solution.

Example :- 0.1 normal NaOH solution prepared, NaOH solution should be standardized by titrating with any primary standard acid.

Error :- Difference between the standard value and observed value.

It is impossible to completely eliminate an error.

They can be classified as:-

a) Determinant Error [cons. Error]

b) Indeterminant Error

Determinant Error

Eg:- Error in titration

Those error which can be avoided or whose magnitude can be determined.

DEPTH OF BIOLOGY

a) Operational Error and personal:-

- When a person is responsible and do not depend on method or procedure.
- It arise because of erratic personal judgement.

Example:- Judgement of color of the solution at the end of titration, wrong reading of weight from balance, wrong level of meniscus from burette, pipette etc.

- This error will vary from person to person and can be minimized by exp. and careful physical manipulation.

b) Instrumental and Chemical error:-

- Electrical instrument are very much prone to determinant error because of fluctuation in voltage etc. Error due to un- adjacent balance use of uncalibrated weight etc.
- Use of low grade chemical is rise to this kind of error.

3. Method error

- It arises due to incorrect sampling and incomplete reaction [97°]

Example- Gravimetric determination

- In this G.D. along with the desire precipitate another substance also get precipitate and which gives error.

DEPTH OF BIOLOGY

Determinant error can further be class into constant and proportional error:-

1. *Constant error:-* It is independent of amount of sample taken.
2. *Prop. Error:-* Mag. of error depends upon the sample size Indeterminant error/Random error

In arises due to uncertainty associated with every physical or chemical measurement.

- They do not have any known cause
- They cannot be corrected or eliminated
- They cannot be predicted
- It is also known as accidental/Random error

Source of error:-

1. *Human Source* :- Qualification and experience of analyst.

Performing the analysis has major impact on error in result

Unexperience person → Error (↑)

Experience person → Error (↓)

2. *Instrument, Apparatus and glassware* :-

Low quality and uncalibrated

Chances of error

DEPTH OF BIOLOGY

3. *Experimental Condition* :- Unfavorable condition result will not obtain.

4. *Constituent used in Analysis* :- If various constituent like stand, solvent, reagent etc.

- Used in analysis are not of desired quality then result will obtain error.

5. *Procedure*:- If the analytical procedure used in analysis is not validate and if validate but not followed carefully then error in result will obtain.

DEPTH OF BIOLOGY

Some of the methods to minimize the error are discussed as follows:-

1. *Instrumental Error* :- Proper calibration should be perform to ensure the performance of equipment.
   - Faculty equipment's should be corrected by experts and rechecked for accuracy of result. If the performance are not satisfied then replacement should be done.
2. *Personal Error* :-
3. *Chemical Error* :- Standard chemicals from authentic source without impurities must be used for analysis. The quality of chemicals and reagents can be checked periodically as per standard guidelines.

DEPTH OF BIOLOGY

4. *Error in Methodology* :- These error can be avoided by following the standard method with proper reference. Continuous monitoring of reaction by skilled person can be employed to minimized error.

5. *Intermediate Errors* :- Since indeterminate error are not predictable, the entire procedure of analysis should be carried out in a well- planned manner considering all factor which affect accuracy of the result.

DEPTH OF BIOLOGY

Pharmacopoeia

- Pharmakon's :- Drug
- Poeia :- make
- Formula or standard required to make a drug.

Pharmacopoeia→It is a government official book which describe the standard of drug test for identification, efficacy and potency to be maintained strictly.

- It contains collection of monographs and published by an authorized body like government a pharmaceutical society.

DEPTH OF BIOLOGY

Monographs → It is a collection of detailed information on a particular drug, chemical name, assay, formula, solubility, identification, pH, dose.

History Of Pharmacopoeia:-

Father of Pharmacy → Claudius Galen

Drug Start → 1870

1881 → Pharmacist Training Start

Pharmacist 1st College → BHU (1937)

B. Pharma Start → 1944 [Punjab College]

USP → 1820 [1st Edition]

B.P. → 1864 [1st Edition]

DEPTH OF BIOLOGY

I.P. → 1955 [1st Edition]

Suppliment → 1960

2nd Edition of I.P. → 1966

Suppliment → 1975

3rd Edition of I.P. → 1985

Suppliment-1 → 1989

Suppliment-2 → 1991

4th Edition of I.P. → 1996

5th Edition → 2007

Addendum → 2008

6th Edition → 2010

Addendum → 2012

DEPTH OF BIOLOGY

7th Edition → 2014 Addendum → 2015, 2016

8th Edition → 2018 Addendum → 2019

Importance of Pharmacopoeia :-

- To maintain the uniformity and control the standard of the drugs available in market.
- Avoid adulterated drug
- Complete information on drug and their dosage form
- Reference for laboratory, industry and academic instruction.

Impurities in Pharmaceutical Substance

Impurity :- It is a undesirable, unwanted element or substance in pharmaceutical that lowers the quality and value of that substance.

Types of Impurities :- According to the ICH [International Council for Harmonisation], impurities are classified as:-

- Organic impurities
- Inorganic impurities
- Residual solvents

DEPTH OF BIOLOGY

Organic impurities :- May arise from starting material by product synthetic intermediates and degradation products.

Inorganic impurities :- May be derived from the manufacturing process and are normally known and identified as reagents, ligands, inorganic salts, heavy metal, catalytic, charcoal etc.

Residual solvent :- Residual solvent are the impurities introduced with solvents.

Source of Impurities in Pharmaceuticals:-

Its various sources of impurities in pharmaceutical substance as follows:-

1. Raw materials
2. Method of manufacturing

a) Reagent used

b) Intermediates products

c) Atmospheric condition

d) Solvents used

DEPTH OF BIOLOGY

3. *Manufacturing hazards*

a) Contamination from matter
b) Contamination by microbes
c) Error in manufacturing
d) Error in storage and packaging

4. *Instability of Products*

a) Chemical instabilities
b) Physical instabilities
c) Reaction with containers
d) Temperature