

Chapter = 07

Analytical Chemistry

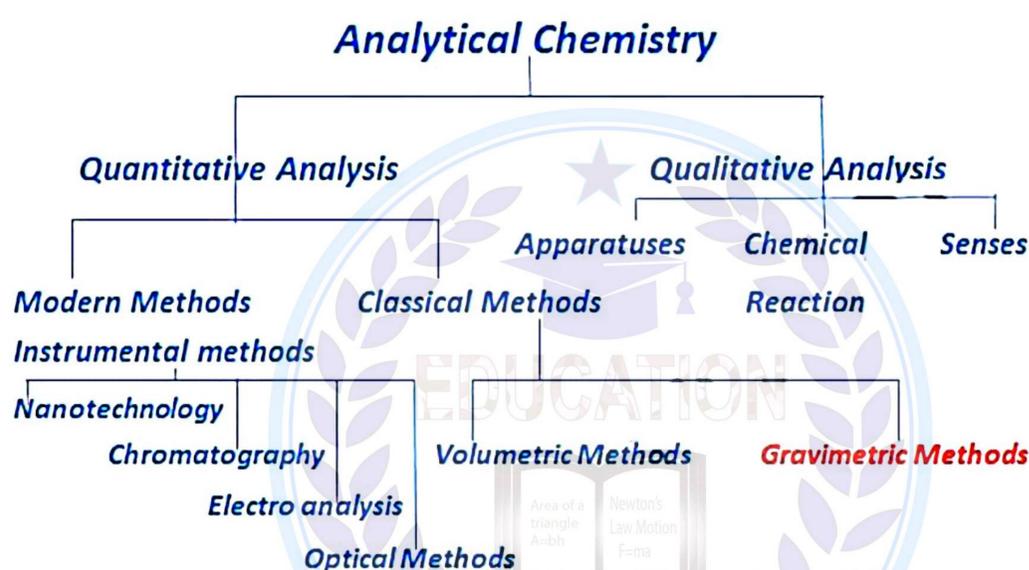
Q1. Define Analytical chemistry, its objective and applications.

The analysis and separation of sample to detect and estimate its components through various techniques and instruments is known as analytical chemistry. 

Objective: The main objective of analytical chemistry is to develop an understanding of analysis of elements and compounds for measurement and problem solving with the help of analytical methods.

Applications: The analytical chemistry is applied in all fields of chemistry such as medicine, clinical laboratories, industries, agriculture, food contamination and environmental protection.

Classification of Analytical Chemistry



Q2. What do you know about Classification of Analytical Chemistry?

Classification of Analytical Chemistry

Analytical chemistry consists of two main types of analysis which are as follows:

(1) **Qualitative analysis:** The identification of elements, ions or compounds present in sample is called quality analysis. The sample may be solid, liquid, gas or a mixture. Qualitative analysis does not measure the quantity of substance but measure the quality of that material. Quantitative analysis is performed by selective chemical reactions or with the use of instrumentation.

Example Chemical test and flame test.

Types of qualitative analysis: Qualitative analysis further divided on the basis of chemical test are as follows.

(i) **Organic qualitative analysis:** It deals with the identification of presence of different classes of organic compounds or functional groups by producing colours in chemical reactions.

Example: Formation of white precipitate by adding silver nitrate (AgNO_3) in dilute nitric acid (HNO_3) indicates the presence of halide ($\text{X}=\text{F},\text{Cl},\text{Br},\text{I}$).

(ii) **Inorganic qualitative analysis:** It deals with the identification of elements. Example: Flame test of copper halide which shows bluish-green colour due to presence of copper. Some other flame test of halide are given in the table.

LiCl	Red Flame
NaCl	Yellow flame
KCl	Light lilac flame
RbCl	Violet Flame
CaCl ₂	Orange Flame
SrCl ₂	Red or Crimson flame
BaCl ₂	Light green flame
CuCl ₂	Blue or green flame

Q3. What is Quantitative analysis?

Quantitative analysis

The determination of how much amount or quantity of one or more substance present in compound or sample is called quantitative analysis. It deals with large number of quantifying methods which are classified as physical or chemical.

Q4. What is Physical methods of analysis?

Physical methods

It measures physical properties such as density, temperature, absorption of light, magnetic influences, colour, and texture.

The physical methods used to measure these properties are Fourier transform infrared spectroscopy (FTIR), atomic emission spectroscopy (AES), trace element analysis and energy dispersive X-ray spectroscopy (EDS).

Q5. How Chemical methods is use to analysis?

Chemical methods

They measure chemical reactions such as precipitation, oxidation or neutralization and are measured by volumetric analysis, gravimetric analysis and combustion analysis.

Q6. What do you know about Parameters? Also give its importance.

Parameters



The parameter is a measurable factor or boundary which defines performance and quality of analytical methods.

Important Parameters

The validation of any analytical method is observed by parameters and various parameters of validation are selectivity, linearity, range, accuracy, precision, and error.

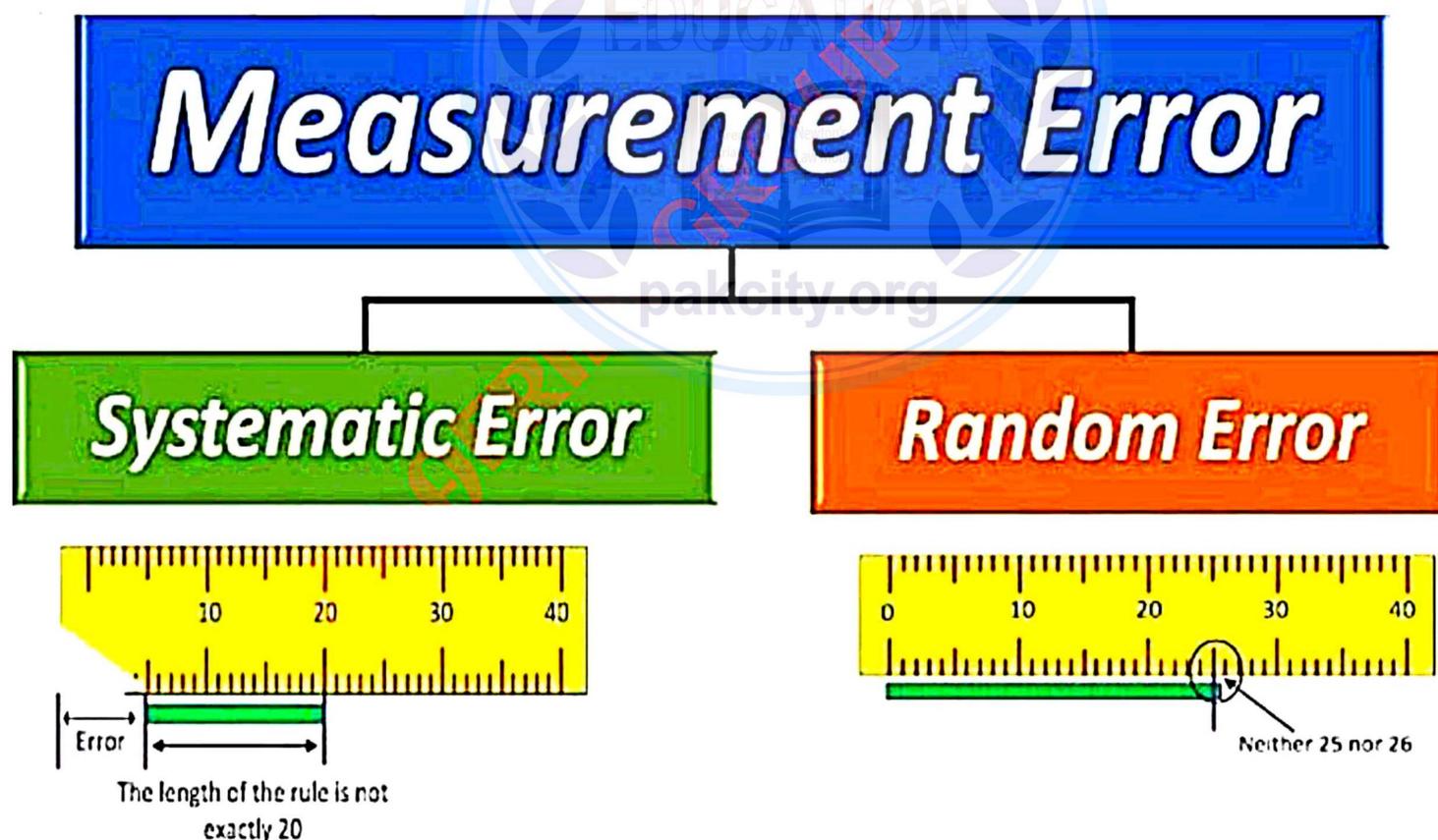
Q7. What do you know about Error? How are they classified?

Error

Error can be defined as numerical difference between observed value and true value.

Classification of errors: Errors in analytical chemistry are classified as systematic and random errors.

1. Systematic errors
2. Random errors



A. Systematic errors or determinate errors

They are caused by defect in analytical method or improper functioning of instrument. Systematic error may be instrumental, observational, environmental and theoretical. Systematic error is also known as determinate error.

B. Random error

Random error may be positive and negative and cannot be eliminated from experiment due to this reason we take many readings and average them.



Q8. What is Accuracy and precision?

Accuracy

Accuracy is an agreement between a measured value and the accepted true value.

Precision

The precision is defined as the degree of agreement between replicate measurements of the same quantity.

Not depend on precision

Accuracy is not dependent on precisions.

Q9. Explain the Different aspects of precision and accuracy with an example.

A measurement can be precise but not accurate, accurate but not precise, neither or both. A measurement system is valid if it is both precise and accurate.

For example

4 Students are performing an experiment to measure the density of aluminum (2.7 g/ml) and note down the following data which shows different aspects of precision and accuracy,

such as measurement of student number 1 is precise because 2.9 is repeating but not accurate because it is not closest to true value.

Measurement of student number 2 is not precise and not accurate because values are not closest to true value and not repeatable.

In the same manner measurement of student number 3 is not precise but accurate due to the closeness of measurement with true value,

while measurement of student number 4 is precise and accurate which may consider a valid measurement system.

Student 1	Student 2	Student 3	Student 4
2.924 g/ml	2.316 g/ml	2.649 g/ml	2.701 g/ml

2.923 g/ml	2.527 g/ml	2.731 g/ml	2.699 g/ml
2.925 g/ml	2.941 g/ml	2.695 g/ml	2.702 g/ml
2.926 g/ml	2.136 g/ml	2.742 g/ml	2.698 g/ml
Precise	Not Precise	Not Precise	Precise
Not Accurate	Not Accurate	Accurate	Accurate

The above example shows that good precision does not assure good accuracy but a valid measurement system needs good precision as well as accuracy.



Q10. What is Classical Method or Wet Chemical Method? Also classified it.

Classical Method or Wet Chemical Method

Classical methods are those analytical techniques which do not use any mechanical or electronic instrument rather than weighing balance. This method is basically related with the chemical reactions between analyte and reagents. It is also known as wet chemical method.

Classification of classical method:

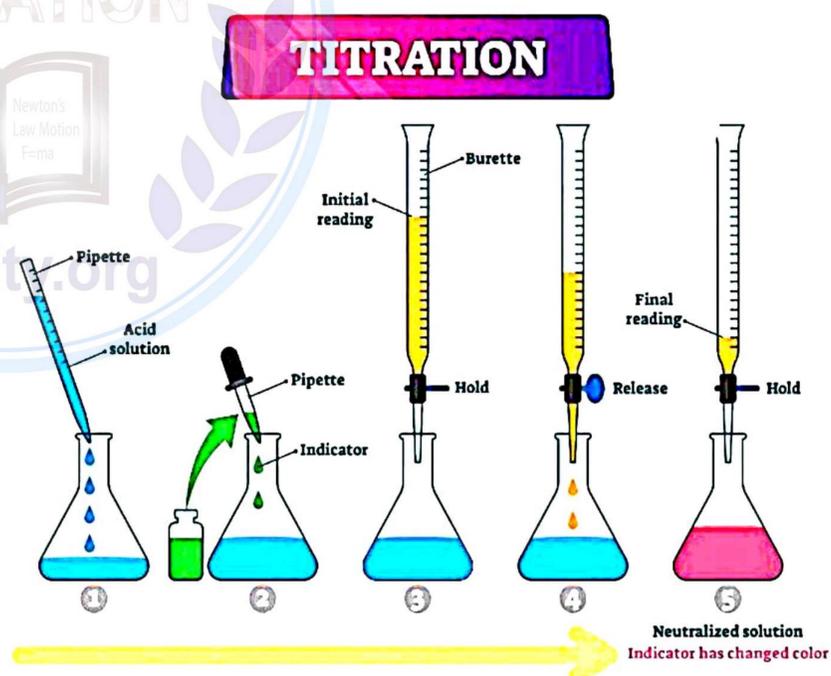
1. Titrimetric Analysis
2. Volumetric Analysis

Q11. Discuss the Titrimetric Analysis or volumetric analysis.

Titrimetric Analysis or volumetric analysis

The titrimetric analysis is used to determine the volume of a solution with known concentration which reacts with the measured volume of solution of a substance quantitatively. The titrimetric analysis is also known as volumetric analysis.

General rule: In this analysis general rule of titration is applied in which volumetric measurement of a reagent takes place which is known as analyte and this analyte completes its chemical reaction with titrant. The general chemical reaction for titrimetric analysis is as follows



Where α is the number of moles of analyte A contained in the sample that reacts with t moles of the titrant T in the titrant solution.

Construction



This reaction is carried out in a flask containing dissolved analyte and indicator while a burette contains titrant solution. An indicator is also added in flask to show the end point of the whole reaction.

Working: Titrant is volumetrically delivered to the flask for reaction. The titration is completed when a sufficient amount of titrant added with analyte for chemical reaction and an equivalence point reached.

Titration: The comparison of volume of a solution of known concentration with the volume of solution of unknown concentration is titration.

Q12. Describe the Gravimetric Analysis.

Gravimetric Analysis

Gravimetric analysis is the oldest and important technique for quantitative estimation in chemical analysis.

Working: In this analysis an amount of analyte is determined by converting the analyte to some product and then weighing it.

For example

you want to determine the amount of chlorine (Cl) present in solution of AgCl then you have to go through following 4 steps for Gravimetric analysis.

- (1) Preparation of a solution with known weight of sample (AgCl).
- (2) Separation of the desired constituent (Cl).
- (3) Weighing separated constituent.
- (4) Computation of amount of separated constituent in the sample.

The gravimetric calculation based on gravimetric factor which converts the grams of the compound into grams of the single element.

Q13. Name the Types of gravimetric analysis

Types of gravimetric analysis

There are four types of gravimetric analysis which are

Physical, Thermos, Precompetitive and Electro gravimetric analysis.

Q14. What do you know about Advanced instrumental methods?

Advanced instrumental methods 

These advanced methods involve usage of instrument for analysis and separation of mixtures and compounds. The methods used as quantitative and quantitative analysis. These analytical advanced instrumental methods include spectroscopy, chromatography, electrochemical methods, ultraviolet and visible spectroscopy, infrared spectroscopy, HPLC, gas chromatography, potentiometry and conductometry.

Q15. What is Spectroscopic method? Also give it application.

Spectroscopic methods

Spectroscopy is the interaction of light with matter.

Application:

1. Spectroscopy is used in physical and analytical chemistry for the identification of substances through the emission or absorption spectrum.
2. The spectroscopy is used to assess concentration or amount of given chemical (atomic, molecular or ionic).

Q16. Name the Measuring device of spectroscopy.

The instruments used for measurement through spectroscopy are called spectrometer, spectrophotometer, and spectrograph.

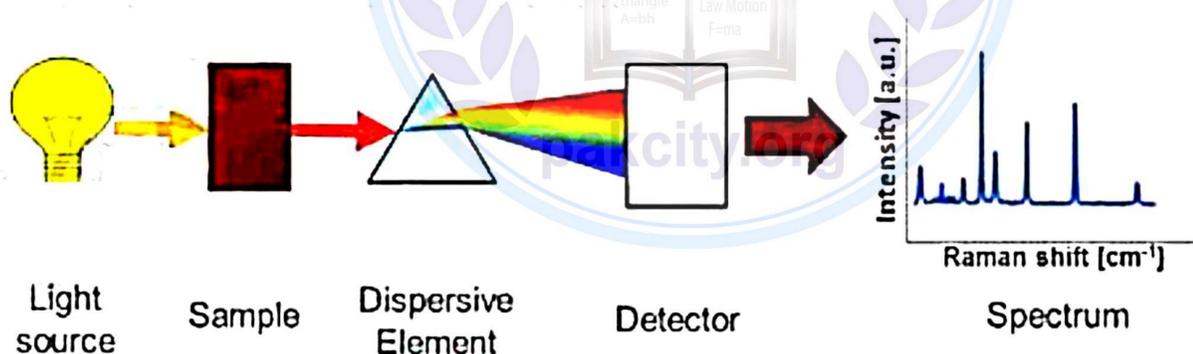


Figure 7.13 Spectroscopic methods

Q17. Name and discuss Types of spectroscopic methods.

Types of spectroscopic methods are given below.

1. Ultraviolet and visible spectroscopy.
2. Infrared spectroscopy.

Ultraviolet and visible spectroscopy

It is a quantitative technique which measures how much a chemical compound absorbs light. This is done by measuring the intensity of light passing through the sample. It is also known as electronic spectroscopy.



Principle: The basic principle of this spectroscopy is interaction between light and matter but here light wavelength is ultraviolet and the process is formation of spectrum due to absorption of ultraviolet light to the chemical compound or sample.

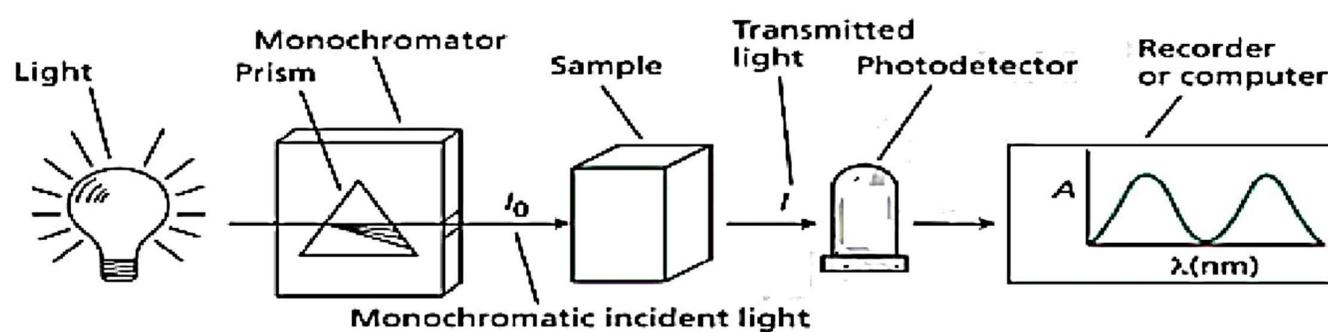


Figure 7.14 Ultraviolet and visible spectroscopy

Infrared spectroscopy or vibrational spectroscopy

Introduction: this technique was introduced in 1950.

Definition: It qualifies and quantifies the information about samples using light whose wavelength is Infrared. It is also known as vibrational spectroscopy.

Properties:

1. It qualifies and quantifies the information about samples in less time and cost effective.
2. It is nonhazardous because no any polluting chemical is required for this analysis.

Uses:

1. It is basically used for specification of functional groups in food products, polymers and industries now a days.
2. It is an effective tool for quality control in different industries.

Q18. What do you know about Infrared radiation?

Definition: Electromagnetic radiations lower in energy than visible radiations are called infra-red radiation.

Wavelength: The ordinary infra-red region extends from 2.5 μm (wavelength) to 15 μm wavelength.

Wavenumber: Wavenumber of infra-red radiation is from 4000 to 625 cm^{-1} wavenumber.

Q19. What is Chromatographic methods? Also list its properties.

Chromatographic methods: Chromatography is the modern analytical technique which is used for the separation of compounds. It also facilitates the purification, isolation and comparison of components of mixture.

Properties:



1. It may be employed with all kinds of volatile and soluble substances, organic and inorganic, polar and nonpolar etc.
2. Chromatography process starts with the mobile phase in which solutes are dissolved in substance and carry to the next stationary phase. The different components of mixture travel from mobile to stationary phase with different speed and retention time.

Q20. Name and discuss the Types of chromatography

Types of chromatography

The main types of chromatography are given below.

1. High performance liquid chromatography (HPLC).
2. Gas chromatography.

High performance liquid chromatography (HPLC)

Definition: It is the technique to separate out the substances. It is also considered as pressure liquid chromatography.

Construction: HPLC instrument consists of a reservoir of mobile phase, a pump, an injector, a separation column, a detector and data acquisition computer.

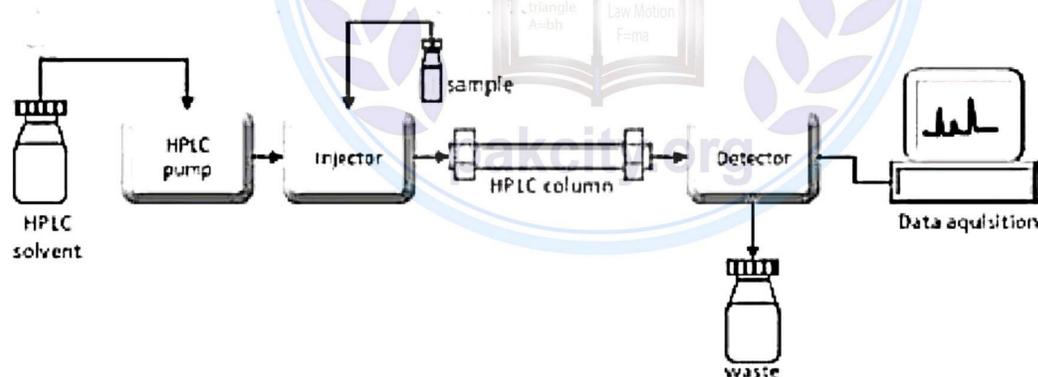


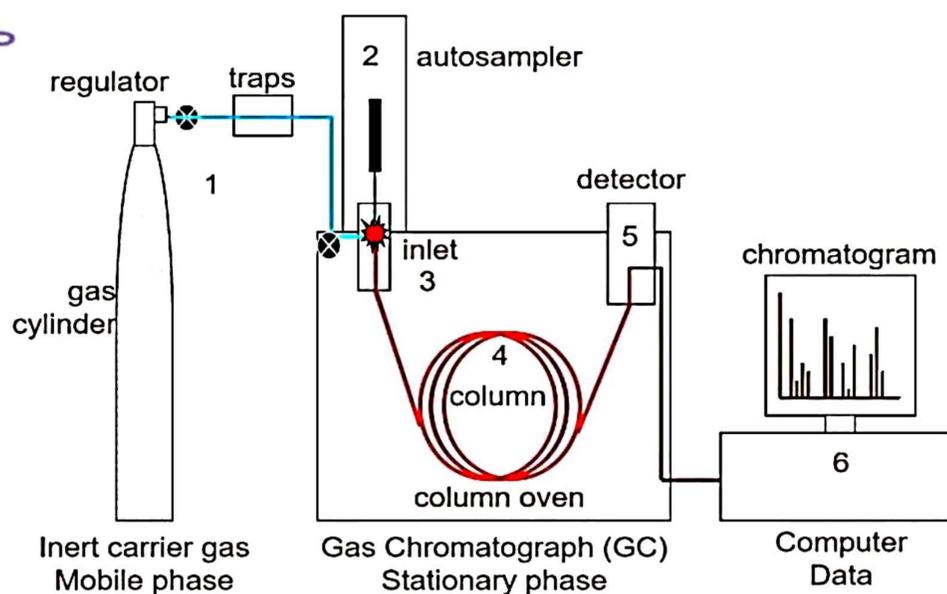
Figure 7.17 HPLC

Working: The mobile phase is pumped through the column packed with the absorbent; hence separation becomes more rapid. The pressure mechanical pump ensures the rapid solvent flow. The flow rate of solvent affects the resolution of sample components. As each component passes through the column, the detector notes its elution and gives signal to the recorder.

Uses: These instruments are used in drug discovery, clinical analysis, cosmetic analysis, pharmaceutical, environmental chemistry and biochemical genetics.

Gas chromatography

Introduction: This method was introduced by John Potter Martin in 1950.



Definition: A gas chromatography is a technique used in analytical chemistry for the separation of gases and volatile liquids.

Principle: This separation takes place by the exchange between a mobile gas phase and a liquid or solid stationary phase.

Construction: The instruments of Gas chromatography consist of Gas cylinder, sample injector, gas chromatograph detector, and data collection device.

Working: Gas is mobile phase and gas cylinder controls the gas passage up to sample injector, which proceeds toward two columned gas chromatograph it is a stationary phase with uniform temperature. When the compound reaches the detector it detects the elution and sends signals to data collection device (computer).

Uses: The gas chromatography used in analysis of inorganic compounds, carbohydrates, proteins, lipids, vitamins, pollutants like benzene, plastic minerals and dairy products.

Q21. Describe the construction and working of Electrochemical methods

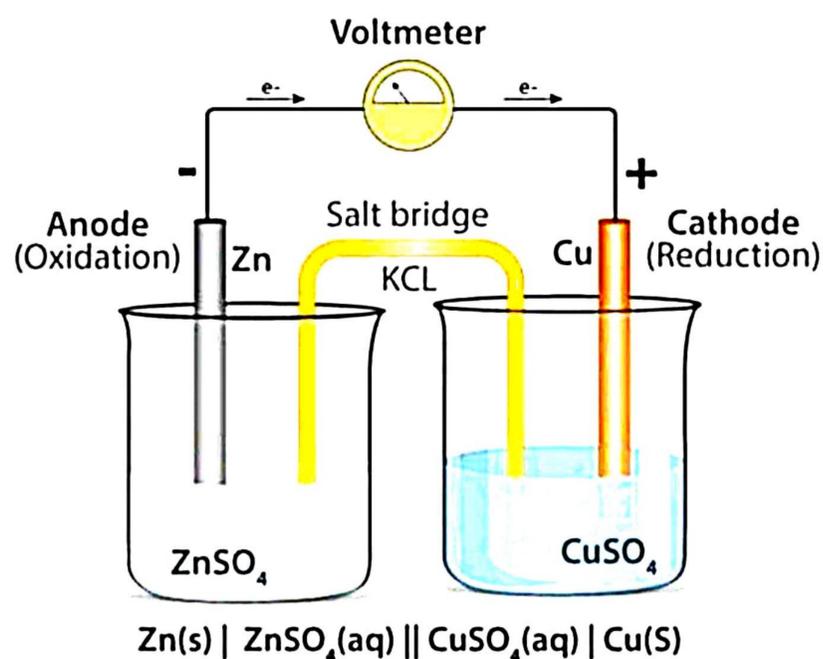
Definition

The electrochemical method is an analytical technique which deals with measurement of potential, charge, electrical quantity or property of a solution.

Construction

The electrochemical analytical method is carried out with the help of electrochemical cell which is shown in the following figure, generally it consists of electrodes named as anode and cathode. Anode possesses negative sign due to liberation of electrons in oxidation reaction and cathode possesses positive sign due to consumption of electrons in reduction reaction.

The electrochemical cells consist of two half cells, both are connected with an electrode (anode and cathode) and each electrode is dipped in electrolytic solution which is $ZnSO_4$ at anode and $CuSO_4$ at cathode.



The half cells are connected by means of salt bridge (NaCl) which provides a platform for ionic connectivity without mixing.

Working: One of half cells loses electrons due to oxidation and other half gains electrons in reduction process. Always remember that when equilibrium phase comes in both half cells the net voltage becomes zero and production of electricity by cell will stop.

Q22. What is Potentiometry? list its uses.

Potentiometry

The potentiometry is a method used in electroanalytical chemistry to find the concentration of solute in solution in potentiometric measurement.

Measuring device

Potential between two electrodes is measured by voltmeter.

Uses

Potentiometric analysis is used in analysis of pollutants in water, pharmaceutical and drugs, quality control in food industry and clinical chemistry.

Q23. What is Conductometry? Give its application.

Conductometry



Conductometry is one of the important analytical techniques which is used in physico-chemical analysis. It can be defined as a technique of analysis which is based on the measurement of electrical conductance.

Measuring device

It is done by the help of conductivity meter.

Application:

1. Degree of dissociation constant can be determined.
2. Solubility of a sparingly soluble salt can be determined.
3. Rate constant of a reaction can be studied.
4. End point of titration can be determined.



S.No.	Classical Method	Instrumental Method
1.	Procedure is simple and accurate.	Procedure is sensitive and technical.
2.	Equipment needed is cheap.	Equipment needed is expensive.
3.	Methods are based on absolute measurement.	Methods are based on liable measurement.
4.	Specialized training is not required.	Specialized training is required.
5.	Accuracy decreases by decreasing amount.	Accuracy depends upon instruments.
6.	Determination is slow.	Determination is very fast.
7.	Large amount of sample is needed.	Small amount of samples can be used.

MULTIPLE CHOICE QUESTIONS

1. The analytical chemistry deals with instruments and methods to _____, identify and quality the matter.
(a) Mix (b) Separate (c) Differentiate (d) Manipulate
2. The sample may be solid, liquid, gas or —- a in qualitative analysis.
(a) Mixture (b) Compound (c) Substance (d) None of these
3. Analysis deals with the identification of presence of functional groups in compounds is.
(a) Physical qualitative analysis (b) Analytical qualitative analysis
(c) Organic qualitative analysis (d) inorganic qualitative analysis
4. Flame test of Copper Halide with bluish-green colour identify the presence of.
(a) Halogen (b) Hydrogen (c) Copper (d) b and c

5. The physical methods used to measure physical properties is called.
- (a) Combustion analysis method (b) Atomic emission spectroscopy method
(c) Volumetric analysis method (d) Gravimetric analysis method
6. The error caused by improper functioning of instrument is:
- (a) Determinant Error (b) In determinant Error
(c) Systematic Error (d) Both a and c
7. An agreement between a measured value and the accepted true value.
- (a) Error (b) Accuracy (c) Precision (d) All of these
8. Spectroscopy is the interaction of light with:
- (a) Liquid (b) Solid (c) Gas (d) Matter
9. The gas is mobile phase in:
- (a) Liquid chromatography (b) Solid chromatography
(c) Gas chromatography (d) None of these
10. It is used to assess concentration or amount of given atomic, molecular or ionic chemical.
- (a) Chromatography (b) Spectroscopy (c) Conductometry (d) Potentiometry



1. Separate	2. Mixture	3. Analytical qualitative analysis	4. Halogen	5. Atomic emission spectroscopy method
6. Both a and c	7. Accuracy	8. Matter	9. Gas chromatography	10. Potentiometry