5th Semester

Branch	Subject Title	Subject Code
B.Sc. (CHEMISTRY-Major)	**INSTRUMENTAL TECHNIQUES IN CHEMISTRY**	**CHM1-151**

Course Objective - Students will learn to design an experiment, select appropriate instrumentation, research and apply laboratory procedures, carry out experiments, troubleshoot instruments, analyze data, and write it all up in a scientific report. It includes techniques such as spectroscopy, mass spectrometry, crystallography, electrochemical analysis, thermal analysis, separations, and microscopy. These methods are employed to study and understand chemical systems, including the molecular structure, concentration, and composition of the analytic.

Course Learning outcomes (CLO) -

By the end of this course students will learn the following aspects of Instrumental Techniques in Chemistry:

- Preparation of standard samples for analysis.
- Instrumentation for analytical methods of Chemistry.
- Instrumentation for various spectroscopic techniques.
- Principles and instrumentation of various electro analytical techniques,
- Instrumentation used in optical methods of analysis.
- Advanced chromatographic techniques

CONCEPT

Unit – 1

Practical Aspects of Chemical Analysis

Analysis of real samples: Choice of analytical method. Analysis of standard samples, preparing standard samples for analysis, moisture in sample, drying the analytical sample, decomposition and dissolution of sample, source of errors in decomposition and dissolution.

Automation in Laboratory: Introduction, classification of analytical methods.Types of instrumental methods. Importance of instruments for analysis. Analog & Digital signals, planising for laboratory automation. An overview of automatic instruments & instrumentation. Good laboratory practices. Instrumental standardization, optimization of procedures.

Unit – 2

Electronic & Vibrational-Rotational Spectroscopy

1. Electronic or Ultra-Violet Visible (UV-Vis) Spectroscopy: Basic principles. Instrumentation and Techniques.

2. Fourier-transform infrared (FTIR) Spectroscopy: Introduction and basic principle of IR spectroscopy, Instrumentation, Working of FTIR Spectrophotometer, Advantages of FTIR Spectroscopy.

3. Raman Spectroscopy: Mechanism of Raman Effect - Quantum theory and classical theory. Instrumentation and techniques. Qualitative treatment of Rotational Raman effect, Effect of nuclear spin, Vibrational Raman spectra, Stokes and anti-Stokes lines, their intensity difference, rule of mutual exclusion.

Unit – 3

Molecular Characterization Techniques

1. Nuclear Magnetic Resonance Spectroscopy: Basic principles of NMR, Instrumentation - Magnet, sweep generator. RF generator, RF receiver, signal recorder, calculation of NMR signals.

2. Electron Spin Resonance (FSR) spectroscopy: Introduction, principle, instrumentation, selection rules, interpretation of Lande's factor 'g'. Hyperfine and super hyperfine coupling.

3. Mass Spectrometry: Theory of mass spectrometry. Principle and operation of mass spectrometer. Ionization techniques- electron impact chemical ionization, electrospray. electrical discharge, laser desorption, fast atom bombardment.

Separation of ions on basis of mass-charge ratio. Analyzers Magnetic-sector, Electric quadrupole and high-resolution multiple-reflection time of flight MR-TOF).

Unit – 4

Atom Characterization Techniques

1. **Flame photometry:** Flame emission spectroscopy, characteristics of flame, instrumentation & working of flame photometer.
2. **Atomic Absorption Spectroscopy (AAS):** Basic principles, Instrumentation, atomizer, monochromator, detector, sensitivity and detection limits. Interferences in AAS and their elimination.
3. **Atomic Emission Spectroscopy (AES):** Principles, Sources for excitation, Instrumentation, Qualitative and quantitative Analysis.

Unit – 5

Electro analytical techniques

1. Polarography: General principles and instrumentation of polarography, half- wave potential, equations for reversible cathodic, anodic and cathodic-anodic waves, analysis of reversible polarographic wave.

2**. Voltametry**: General principles and instrumentation. Cyclic voltammetry. Linear-scan voltammetry, Pulse voltammetric methods, Voltammetry with ultra-micro electrodes, stripping methods.

3. Amperometry: Principles and amperometric titration techniques-Dropping mercury electrode, rotating platinum microelectrode

4. Potentiometry: Introduction, reference and indicator electrodes, ion selective electrodes. Instrumentation and measurement of electro motive force of cell (EMF). Potentiometric titrations.

5. Conductometry: Principle, measurement of conductance, conductometric titrations.

Unit – 6

Optical and Advanced Chromatographic Techniques

1. Polarimetry: Polarimeter, optical rotations, measurements of optical rotation.

2. Refractometry: Principle of refraction, Snell's law, Construction & working of refractometer.

3. Gas Chromatography (GC): Theory, Instrumentation-description of equipment and different parts, columns (packed and capillary columns).

Detector specifications, Thermal conductivity detector, Flame tonization detector, electron capture detector, nitrogen-phosphorous detector or thermionic specific detector (TSD), photo ionization detector, Programmed temperature gas chromatography.

4. High Performance Liquid Chromatography (HPLC): Theory, Instrumentation, description of the different parts of the equipments, stationary phases (columns), mobile phase, detectors, UV detector, refractive index (RI) detector, Fluorescence detector, Photo Diode Array detector, Evaporative Light Scattering Detector (ELSD), conductometric detector and electrochemical detector.

Practical's

1.Spectrophotometry

1. Determination of concentration of ferric ions in ferric salicylate complex spectrophotometrically.
2. Simultaneous determination of chromium and manganese by spectrophotometry
3. Determination of sulphate and phosphate by spectrophotometry.
4. Spectrophotometric determination of pK value of an indicator.
5. Determination of magnesium and calcium in tap water by flame photometry.
6. Spectrophotometric determination of the stoichiometry and stability constants of complexes.

2.Refractometry & Polarimetry

1. Verification of law of refraction of mixtures (e.g. glycerol and water) using Abbe's refractometer.
2. Determination of the specific rotation of a given optically active compound by polarimetry.
3. Determination of specific rotation of glucose and fructose by polarimetry
4. Determination of the enzyme catalyzed inversion of sucrose by polarimetry
5. Determine the concentration of a solution of an optically active substance by polarimetry.

3.Potentiometry

1. Determination of the standard EMF and the standard free energy change of Daniel cell potentiometrically,
2. Potentiometric titration of a given hydrochloric acid solution with an alkali solution. Potentiometric titration of given ferrous sulphate solution with potassium dichromate .
4. Determination of solubility product of a sparingly soluble substance.

4.Conductometry

1.Conductometre titration of hydrochloric acid with sodium hydroxide.
2. Conductometric titration of hydrochlonic seid with ammonium: hydroxide.
3. Conductometric titration of acetic acid with ammonium hydroxide.

5.Polarography

1. Determination of the half wave potential of metal ion by polarography.
2. Determination of the amount of Cd(II) ions in an unknown solution by polarography.