

CA H 512: ELECTROANALYTICAL AND THERMOANALYTICAL TECHNIQUES

Course Outcomes

- Student will understand the principles of electrochemistry and applications of electromotive force.
- Acquire knowledge on chemical and biosensors and their analytical uses.
- Gather knowledge about polarographic and voltammetry techniques and their applications.
- Demonstrate a systematic understanding of the key aspects of coulometry, amperometry and electrogravimetry and their analytical applications.
- Understand the theory and applications TGA, DTA and DSC.

UNIT-I: Basic Concepts in Electrochemical Techniques

[12 Hours]

Electrochemical cell: Components, conduction, reactions, liquid junctions, salt bridge representation. Faradaic and non-faradaic currents. Reversible and irreversible cells.

Electrode potential: Nature, measurement, sign convention, effect of concentration. Standard electrode potential, calculation limitations.

Cell potential: Thermodynamics, liquid junction potential, effect of current, ohmic potential.

Polarization: Sources, overvoltage, concentration polarization, mechanism of mass transport,

Potentiometric methods: Reference electrodes-Calomel, silver/silver chloride and hydrogen electrodes, potentiometric titrations and applications.

Metallic electrodes: Electrodes of first, second & third kind.

Membrane electrodes: Classifications, properties, principle, design of ion selective electrodes, membrane potential, selectivity, crystalline liquid membrane and enzyme electrodes.

Glass electrode: Composition, hygroscopicity, asymmetry potential, acid and alkali errors.

Conductometric methods: Definitions, conductance measurement, conductometric titrations and applications.

UNIT-II

[12 Hours]

Polarography and voltammetry: Theory of classical polarography, polarographic measurements, polarograms, polarographic currents, current and concentration relationship, factors influencing diffusion currents, Half wave potential, Oxygen interference, Advantages and limitations. Modified voltammetric methods, pulse polarography, fast, linear-sweep polarography, first & second order polarographic techniques, organic polarography.

Voltammetric principles: Voltammetry at solid electrodes-Hydrodynamic voltammetry, triangular voltammetry or cyclic voltammetry.

Stripping analysis: Principle, methodology, electrodes & cell design, applications and determination of lead in water by voltammetry.

Chemical sensors and biosensors: Sensors, electrochemical sensors, optical sensors, thermal and mass-sensitive sensors, determination of glucose in blood by biosensor, determination of copper (I) in water using anodic stripping voltammetry.

UNIT-III

[12 Hours]

Coulometry: Principle, constant current and controlled potential coulometry. Coulometric titrations- Principles, apparatus, comparison of coulometric titrations with conventional titrations, automatic coulometric titrations and applications.

Amperometry: Principle, titrations, advantages and limitations. Applications of biamperometric titrations.

Electrogravimetry: Faraday's laws of electrolysis, requirements, decomposition potential and its significance. Over voltage and factors influencing overvoltage. Effect of experimental variables- Physical and chemical. Electrolysis at constant current and at controlled potentials, applications.

Chrono Methods: Basic concepts, methodology and application of chronopotentiometry, chronoampermetry and chronocoulometry.

UNIT IV: Thermoanalytical Techniques

[12 Hours]

Thermogravimetric analysis (TGA): Types, principle and method. Automatic thermogravimetric analysis, instrumentation, types of recording thermo balances, sample holders, factors affecting results. Applications-Purity and thermal stability, evaluation of correct drying temperature, analysis of complex mixture and determination of kinetic parameters of thermal degradation, isothermal analysis.

Differential thermal analysis (DTA): Principle of working, theory and instrumentation. Simultaneous TA-TGA curves, factors affecting results. Applications-Analysis of the physical mixtures and thermal behaviour study, determination of melting point, boiling point and decomposition point. Differences between TGA and DTA.

Differential scanning calorimetry (DSC): Principle, differences between DTA and DSC. Instrumentation, applications studies of thermal transitions and isothermal crystallization. Pharmaceutical industry for testing the purity of the samples.

Thermometric titration: Introduction, apparatus and applications (Acid-base, precipitation, complexation, redox and non-aqueous titrations). Chemiluminescence methods.

References

1. Principles and Practice of Analytical Chemistry, 3rd edn., F. W. Fifeild and Kealey, Blackwell Science Ltd. (2000).
2. Analytical Chemistry Principles, 2nd edn., J. H. Kennedy, Saunders College Publishing, California (1990).
3. Modern Analytical Chemistry, David Harvey, McGraw Hill, New Delhi (2000).
4. Introduction to Instrumental Analysis, 2nd edn., R. D. Braun, Pharm. Med. Press India (2012).