

Andhra University
Department of Inorganic and Analytical Chemistry
M.Sc. (Final) Chemistry Syllabus for 4th Semester
Specialization - Analytical Chemistry
Paper-1: Separation Methods – II
(Effective from 2018-19 Admitted batch)

Unit – I Chromatography - 5

- (a) ***Ion Exchange:*** principles of ion-exchange systems, synthetic ion-exchange resins, properties of anion and cation exchange resins, ion-exchange mechanism, ion-exchange equilibria, selectivity, ion-exchange capacity, applications of ion-exchangers in different fields.
- (b) ***Ion exchange chromatography:*** Principle, Equipment, Application Specifically Separations of Lanthanides, Actinides, amino acids.
- (c) ***Ion chromatography:*** principles of separation, instrumentation, detectors, separation of cations and anions, applications in the analysis of water and air pollutants.

Unit – II Chromatography – 6

Liquid-liquid partition chromatography: principle supports, partitioning liquids, eluents, reverse phase chromatography, apparatus and applications

High performance liquid chromatography: Theory, Instrument description of the different parts of the equipment, columns, detectors-UV detector, refractometric detector, Fluorescence detector, Diode Array detector, applications in the separation of organic compounds, names of other detectors used their Principles and Applications.

LC-MS – Introduction – Instrumentation – liquid chromatograph – Mass spectrometer Interface – Instrumental details – Processing LC-MS data – ion chromatograms – Library searching – Quantitative measurements.
Sample preparation – selected ion monitoring. Application of LC-MS for Drug analysis, Environmental samples and others.

Unit – III Sampling of Solids, Liquids and Gases

Sampling: Basis of sampling, purpose of sampling, homogeneous and heterogeneous samples, statistical criteria for good sampling, sample size, sampling unit, gross sample, laboratory sample.

Sampling of Solids: Cone and Quartering method, Long pile and alternative shovel method, precautions in preservation of solid samples, sampling of metals and other solids rods, wires, sheets, plates, especially Gold, Silver, Iron and other metals.

Sampling of different types of liquids: different sampling techniques, sampling of drinking water, industrial effluents, precautions in sampling and preservation of collected liquid samples.

Sampling of gases: sampling and Pre-concentration by adsorption or absorption method, instantaneous monitoring, sampling in samplers and subsequent monitoring, different types of gas samplers, precautions in preservation of samples, systematic sampling and random sampling.

Unit – IV Importance of Analytical chemistry & Solvent Extraction

(a) Importance of Analytical Chemistry to Industrial Research: Importance of Qualitative and Quantitative analysis in research and development, industries and other branches of science.

Development and validation of an analytical method, units, concentrations, calculations, standards, chemical reactions, expressions of concentrations, importance of separation methods with examples.

(b) Solvent Extraction: principles and processes of solvent extraction, Distribution Law and Partition coefficient, nature of partition forces, different types of solvent extraction systems – Batch extraction, Continuous extraction, Counter current extraction, solvent extraction systems, applications in metallurgy, general applications in analysis and pre-concentration, special extraction systems like crown ethers, super fluid and surfactant extractions-examples.

Text books:

1. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
2. Separation methods, M.N Sastri, Himalaya Publishing Company, Mumbai

Reference books:

1. E. Helfman, Chromatography, Van Nostrand, Reinhold, New York
2. E. Lederer and M. Lederer, Chromatography, Elsevier, Amsterdam.
3. Chemical separation methods, John A Dean, Von Nostrand Reinhold, New York
4. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
5. E.Stahl, Thin layer chromatography, Academic Press, New York
6. James, G.Tartor (Ion chromatography)

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Paper - IV: Instrumental Methods of Analysis -II
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Unit – I: Atomic Spectroscopy

(a) **Atomic Absorption Spectroscopy(AAS):** theory-atomic line width, line broadening, sample introduction techniques-nebulization, electrothermal, *direct insertion, hydride generation laser ablation, sputtering, arc and spark ablation*; instrumentation-flame AAS and non-flame / furnace AAS, **atomization techniques:** flame, electrothermal, **glow- discharge atomization, hydride atomization, cold-vapour atomization and Inductively coupled plasma**, resonance line sources, hollow cathode lamp, sensitivity and detection limits in AAS, chemical and spectral interferences, applications with special reference to analysis of trace metals in oils, alloys and toxic metals in drinking water and effluents

(b) **Flame photometry: principle of flame photometry**, theory, instrumentation, combustion flames, detectors, and analysis of Na, K, Ca, Mg, Li

(c) **Inductively coupled plasma Emission spectroscopy and Inductively coupled plasma Mass spectrometry (ICP-AES, ICP-MS): principle of AES**, instrumentation, plasma, AES detectors, quadrupole mass spectrometers, difference between the two detectors, analysis methods for liquids and solids, applications in the analysis of trace and toxic metals in water, geological and industrial samples.

Unit – II Thermal methods of Analysis

(a) Thermo gravimetry-theory, instrumentation, applications with special reference to $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{CaC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, CaCO_3 , $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$

(b) Differential thermal analysis-principle, instrumentation, difference between TG and DTA - applications with special reference to the clays and minerals, coals (fuels)

(c) Differential scanning calorimetry-principle, instrumentation, applications to inorganic materials like chlorates and per chlorates, ammonium nitrate, organic compounds and Drugs.

Unit- III : Electro analytical Methods of Analysis - 1

(a) **Voltametry and polarographic analysis :** Classification of voltametry, principle of polarography, residual current, migration current, diffusion current, half-wave potential, Ilkovic equation, instrumentation, Dropping mercury electrode (DME), advantages and disadvantages of DME, DC polarography, AC polarography, Pulse polarography-Normal pulse, triangular pulse and squarewave pulse; qualitative and

quantitative analysis of inorganic ions-Cu, Bi, Pb, Cd, Zn, AC polarography, pulse polarography

(b) **Anode stripping voltametry:** principle, instrumentation, Hanging mercury drop electrode, application in the analysis of Pb and Cd in environmental samples, principle of cathode stripping voltametry.

(c) **Coulometric analysis:** principles of coulometric analysis with constant current, coulometric analysis with controlled potential, applications of coulometric methods for the analysis of cations-As (III), Fe (II) and I^- and S^{2-} by using I_2 liberations and Ce^{4+} liberation in solutions

Unit – IV Electro Analytical and Radio chemical methods of analysis - 2

(a) **Ion Selective Electrodes:** reference electrodes - hydrogen electrode, calomel electrode, silver chloride electrode; indicator electrodes – hydrogen and glass electrodes, theory of membrane potentials and liquid junction potentials, types of ion selective electrodes, basic properties, potentials and construction, calibration of ion selective electrodes, ion selective electrodes with fixed membrane sites, silver, lead, cadmium, sulfide, fluoride, cyanide and glass electrodes, applications in the analysis of air and water pollutants, principles of liquid membrane, gas sensing and enzyme based electrode

(b) **Radio chemical methods of analysis:** detection and measurement of radioactivity, introduction to radioactive tracers, applications of tracer technique, isotope dilution analysis - applications, activation analysis – application, advantages and disadvantages, radio carbon dating technique

Text Books:

1. Instrumental methods of analysis – H.H Willard, Meritt Jr. and J.A Dean
2. Principles of instrumental analysis – Skoog and West
1. Vogels Textbook of Quantitative Inorganic analysis – J. Basset, R.C Denney, G.H Jefferey and J.Madhan
2. Instrumental methods of analysis – B.K Sarma, Goel Publishing House, Meerut
3. Instrumental methods of Analysis – Chatwal and Anand
4. Instrumental methods of Analysis – Ewing

Reference Books:

W.Wendtlandt, Thermal Analysis, John Wiley Sons, New York