

SEMESTER –VI
INSTRUMENTAL METHODS OF ANALYSIS
THEORY

Program: B.Sc.
Course Code: U20/CHE/DSE/601
Course: DSE 1
No. of Credits: 3

Max. Hours: 45
Hours per week: 3
Max. Marks: 100

COURSE OBJECTIVES:

- To understand the importance of separation techniques such as solvent extraction and to explore the principles and procedures of chromatographic techniques including paper, thin layer,
- To learn the principles of column, ion exchange chromatography, HPLC and GLC.
- To delve into the principle and instrumentation of UV/Visible spectrophotometry, and its application to the quantitative analysis of various ions.
- An approach towards the principle and application of electroanalytical analysis.

COURSE OUTCOMES:

- CO1:** Understand various separation techniques and choose the most appropriate analytical technique for a variety of samples.
- CO2:** Explain the theoretical principles of various separation techniques in chromatography and their typical applications.
- CO3:** Interpret the theoretical principles of selected instrumental methods with spectrophotometric methods.
- CO4:** Review and assessment of electro analytical methods

INSTRUMENTAL METHODS OF ANALYSIS**MODULE 1: CHROMATOGRAPHY I****(11 Hrs)**

Solvent Extraction- Principle, Methods of extraction: Batch extraction, continuous extraction and counter current extraction. Application – Determination of Iron (III).

Chromatography: Classification of chromatographic methods, principles of differential migration, adsorption phenomenon, nature of adsorbents, solvent systems.

Thin layer Chromatography (TLC): Advantages, preparation of plates, development of the chromatogram, Detection of the spots, factors effecting R_f values and applications.

Paper Chromatography: Principle, choice of paper and solvent systems, development of chromatogram – ascending, descending, radial and two dimensional chromatography and applications.

MODULE 2: CHROMATOGRAPHY II**(11 Hrs)**

Column Chromatography: Principle, Types of stationary phases, Column packing – Wet packing technique, Dry packing technique. Selection criteria of mobile phase (solvents) for eluting, polar, non-polar compounds and its applications.

Ion exchange chromatography: Principle, cation and anion exchange resins, its application in separation of ions.

Gas Chromatography: Theory and instrumentation (Block Diagram), Types of stationary phases and carrier gases (mobile phase).

High performance liquid chromatography: Theory and instrumentation, stationary phases and mobile phases. Analysis of paracetamol.

MODULE 3: COLORIMETRY AND SPECTROPHOTOMETRY**(12 Hrs)**

General features of absorption – spectroscopy, transmittance, absorbance, and molar absorptivity. Beer Lambert's law and its limitations, difference between Colorimetry and Spectrophotometry.

Instruments – Single beam UV- Visible Spectrophotometer, Double beam UV- Visible Spectrophotometer. Lamps used as energy sources. Verification of Beer's law. Estimation of iron in water samples by thiocyanate method. Estimation of (i) Chromium and (ii) Manganese in steel.

IR Spectrophotometer: Principle, Sources of Radiations, Sampling, Block diagram of FT-IR Spectrophotometer.

MODULE 4: ELECTROANALYTICAL METHODS**(11 Hrs)**

Types of Electroanalytical Methods.

I) Interfacial methods – a) Potentiometry: Principle, Electrochemical cell, Electrodes- (i) Indicator and (ii) Reference electrodes – Normal Hydrogen Electrode, Quinhydrone Electrode, Saturated Calomel Electrode. Numerical Problems. Application of Potentiometry – Assay of Sulphanilamide

b) Voltametry – three electrode assembly; Introduction to types of voltametric techniques, micro electrodes, Over potential and Polarization.

II) Bulk methods – Conductometry, Conductivity Cell, Specific Conductivity, Equivalent Conductivity. Numerical Problems. Applications of conductometry. Estimation of Cl⁻ using AgNO₃. Determination of Aspirin with KOH.

Text Books:

1. David Krupadanam, *Analytical Chemistry*, Universities Press (India) Limited.
2. S. M. Khopkar, *Basic Concepts Of Analytical Chemistry*, New Age International Publishers.
3. Gurdeep R. Chatwal, Sham K. Anand, *Instrumental Methods Of Chemical Analysis*, Himalaya Publishing House.

Reference Books:

1. D.A. Skoog, F.J. Holler, T.A. Nieman, *Principles Of Instrumental Analysis*, Engage Earning India Edn.
2. D. A. Skoog, D.M. West, F.J. Holler, *Fundamentals Of Analytical Chemistry* 6 Th Edn., Saunders College Publishing, Fort Worth (1992).
3. Cooper, T.G. *The Tools Of Biochemistry*, John Wiley And Sons, N.Y. USA.16, 1977.
4. Vogel, A. I. *Vogel's Qualitative Inorganic Analysis* 7th Edn, Prentice Hall.
5. Vogel, A. I. *Vogel's Quantitative Chemical Analysis* 6th Edn, Prentice Hall.
6. Gary D. Christian, *Analytical Chemistry* 7th Edition. (2004).

INSTRUMENTAL METHODS OF ANALYSIS**MODEL QUESTION PAPER****THEORY****Course Code: U20/CHE/DSE/601****Max. Marks: 60****Credits: 3****Max. Time: 2 Hrs****SECTION – A****I. Answer the following****4 x 10 = 40 M**

1. Explain the principle of paper chromatography and development of chromatogram by four methods. (CO1) 10M

OR

2. a) Explain the Craig's counter current process of solvent extraction(CO2) 5M
b) Write a short note on Soxhlet extraction technique. (CO1) 5M
3. a) Explain the theory involved in Gas chromatography and draw the block diagram. (CO2) 5M
b) Give the analysis of paracetamol by HPLC (CO3) 5M

OR

4. Describe the principle of Ion exchange chromatography. Give an account of cation and anion exchange resins.(CO1) 10M
5. a) Estimation of iron in water sample samples by thiocyanate method.(CO3) 5M
b) Explain the instrumentation of double beam spectrophotometer. (CO3) 5M

OR

6. Explain the Principle, Sources of Radiations, Sampling technique in IR spectrophotometry. (CO3) 10M
7. a) Describe the working of Calomel electrode with a neat diagram (CO4) 5M
b) How chloride ions are estimated using silver nitrate by conductometry.(CO4) 5M

OR

8. Calculate the EMF of a cell initially of an acid- base titration where 25ml 0.1 M of HCl is titrated potentiometrically against standard 0.1 M NaOH using hydrogen electrode as indicator electrode and saturated calomel electrode as reference electrode. What would be the EMF after the addition of 20, 25 and 30 ml of NaOH solution? (CO4) 10M

SECTION – B**II. Answer any FOUR.****4 x 5 = 20 M**

9. Write any two applications of TLC (CO2)
10. Give an account of different types of column packing. (CO1)
11. Differentiate between Colorimetry and Spectrophotometry.(CO3)
12. Define Molar absorption coefficient and transmittance (CO3)
13. Write a short note on three electrode assembly.(CO4)
14. What are the different types of electrodes used in potentiometry.(CO4)