## **SEMESTER-II**

# 1. Instrumental Techniques for Chemical Analysis

Time: 3 Hrs. M.M. 100 marks

Note: The paper will be divided into two sections.

Section-A M.C.Q.50 (10 from each section) Total-50 marks

**Section-B:** Two questions are from each unit will be asked with internal choice and the candidate is required to attempt five questions. **Total-50 marks** 

#### Unit – I

**Ultraviolet-visible Spectroscopy**: Introduction, energy of electronic excitation, absorption laws, chromophores and auxochromes, solvent effects on UV absorption, conjugated dienes, The woodword Fieser rules for dienes, enones.

**IR Spectroscopy**: Introduction, force constants, stretching and bending modes, and their energy, absorption by various functional groups, finger print region, overtones, combination tones, Fermi resonance, factors influencing vibration frequencies, variation in the carbonyl frequencies, introduction to FTIR spectroscopy.

### Unit – II

**Nuclear Magnetic Resonance Spectroscopy**: Introduction, equivalent and non-equivalent protons, chemical shifts, factors affecting chemical shifts, spin-spin coupling, splitting patterns (AX, AB, AMX, ABX), spin decoupling, deuterium labelling, chemical exchange, coupling constants (geminal, vicinal, long range), NOE, shift reagents.

<sup>13</sup>C NMR Spectroscopy: Introduction, <sup>13</sup>C chemical shifts, off resonance spectra, <sup>13</sup>C-<sup>1</sup>H and <sup>13</sup>C-<sup>13</sup>C coupling interpretation, introduction to FTNMR spectroscopy, interpretation of <sup>13</sup>C spectra of simple molecules.

#### Unit\_ III

Mass Spectroscopy: Electron impact line diagram of mass spectrometer, molecular ion base peak, metastable ion, doubly charged ion, nitrogen rule, isotopic abundance and calculation of molecular formula, isotopic profile of helogen compounds, fragmentation patterns simple cleavage, retro-diels Alder, hydrogen transfer, rearrangement like scrambling, ortho effect, McLafferty rearrangement. Fragmentation pattern of simple aliphatic and aromatic compounds. Elementary idea of chemical ionization and negative ion mass spectrometry.

**Solvent Extraction:** Distribution law, techniques for solvent extraction, classification of solvent extraction systems, factors favouring extraction, application of extraction in analytical methods.

**Chromatography:** Classification, theories of chromatographic paper, column, thin layer, ion exchange, gas and HPLC, GPC.

**Polarography:** Principle and instrumental set-up of polarography, concepts and expressions of diffusion current, half-wave potential, residual current, dropping mercury electrode, current potential curve and reversible reactions.

#### UNIT – V

**Thermoanalytical Methods:** Theory, instrumental requirement and methodology, applications of thermo gravimetric analysis (TGA), differential thermal analysis (DTA) and differential scanning calorimetry (DSC). Application of DSC to determination of drug purity, micro thermal techniques and their applications.

# **Books Recommended:**

- 1. Spectral Analysis of Organic Compounds, Crewell Runquist and Campbell.
- 2. Spectroscopic Methods in Organic Chemistry, Williams and Fleming.
- 3. Organic Spectroscopy An Introduction, Dyke, Floyd, Sainsbury and Thiobalt.
- 4. Spectroscopic Identification of Organic Compounds, Silverstein, Bassler and Morril. Organic Spectroscopy, William Kemp.
- 5. "Vogel's Quantitative Inorganic Analysis", Bassette & Coworkers, Longman's Green & Co.
- 6. "Principles of Instrumental Analysis", D.A. Skoog, 4<sup>th</sup> Ed. (1992).
- 7. "Instrumental Methods of Analusis" M.H. Willard, L.L. Merrit, J.A. Dean and F.A. Settle, 7<sup>th</sup> edn. (1988).
- 8. "Principles of Polargraphy", R.C. Kapoor & B.S. Aggarwal, Wiley Eastern Ltd.
- 9. "Fundamental of Molecular Spectroscopy", C.N. Banwell, McGraw Hill (1983).
- 10. "Thermal Analysis", W.W. Wndlanth and L.W. Collins, Dauden, Hutechim and Rose, 7<sup>th</sup> Ed. (1988).
- 11. "Principle and Practice of Analytical Chemistry", F.W. Fitfield and D. Kaley, Blackie Academic & Professional, 4<sup>th</sup> edn. (1995).
- 12. Basic Concepts of Analytical Chemistry: S. M. Khopkar.
- 13. Spectrometric Identification of Organic Compounds: Silverstein.