Course code	Course Title	L	T	P	C
PHD19002	Modern methods of analytical techniques	4	0	0	4

Unit 1: Introduction

Evaluation of analytical data-types of errors in analytical results; Determinate and indeterminate errors and their effect on precision; significant figures and computation. Elemental analysis: molecular mass and formula determination; Index of hydrogen deficiency – rule of thirteen.

Unit 2: Separation techniques

Introduction, principles practice of partition and adsorption chromatography, Gas-liquid chromatography – HPLC - gel chromatography – affinity, Ion exchange and molecular sieve chromatography, Energy and electromagnetic spectrum; absorption and emission spectroscopy – a comparison; molecular formula and structural correlation.

Unit 3: Spectrometric identification of organic compounds – part 1

(a) Ultraviolet – Visible spectroscopy: The nature of electronic excitations – type of radiation absorbed and its effect on the molecule; Electronic energy levels – orbitals involved in electronic transitions – chromophore concept; Laws of light absorption: Beer – Lambert's law and its quantitative application and limitations; Sources of UV radiation – sample and reference cells used – solvents and solutions – solvent effects – vacuum UV Region; bathochromic, hypsochromic, hyperchromic and hypochromic shifts; effect of conjugation. Woodward - Fischer rules for calculation of λ_{max} of dienes (excluding polar group substituents) – Woodward rules for enones (polar substituents –OH, $–OCH_3$, –Cl, –Br); Fischer – Kuhn rules for determining λ_{max} and ε_{max} of polyenes. Use of shift reagents in U.V. spectral analysis, with examples chosen from chemistry of naturally occurring flavonoids- where such methods have been particularly successful.

(b) Infra-Red spectroscopy: Theory of IR absorption process, IR spectrum – complexities; Fundamental absorptions, overtones, combination bands, difference bands, Fermi resonance bands; IR spectrum as a molecular finger print; modes of stretching and bending – calculation of vibrational frequencies – Hooke's law; Sampling techniques – infrared sources, monochromators, detectors used; factors influencing vibrational frequency – hydrogen bonding, electronic effects, inductive and mesomeric effects – bond angles and field effects. Application of IR spectroscopy: Analysis of IR spectral data, identification of the following functional groups: alkanes, alkenes, alkynes, aromatic compounds, carbonyl compounds – (amides, carboxylic acids, ketones, aldehydes, esters, anhydrides, acid chlorides), hydroxy compounds (alcohols, phenols), ethers, nitrogen compounds (amines, nitriles). I.R.spectra with regards to study of hydrogen bonding, and resonance effects in organic compounds.

Unit 4: Spectrometric identification of organic compounds – part 2

(a) Nuclear Magnetic Resonance: Theory of NMR – nuclear spin states, nuclear magnetic moment, mechanism of absorption of energy (resonance), population density of nuclear spin states – precessional (Larmor) frequency. Chemical shift and its measurement: Internal standards in NMR, NMR spectrometer, units used in NMR spectroscopy; factors influencing chemical shift – electronegativity, shielding and de-shielding – Van der Waals de-shielding, magnetic anisotropic effects in alkene, alkynes, carbonyl compounds (ketones and aldehydes) – ring protons. Choice of solvents for NMR, position, intensity, multiplicity of peaks, integrals in PMR; theory of spin-spin coupling, spin-spin splitting tree (proton only); coupling constants – importance; Pascal's triangle; first-order spectra – AX, AX_2 and AX_3

coupling cases; magnetically equivalent protons – coupling constants in aliphatic, aromatic and alkene systems; detailed analysis of NMR spectral data of organic compounds; Illustration by means of solving problems. Use of paramagnetic shift reagents. NOE effect, FT NMR its advantage. Double resonance, with particular reference to spin - spin decoupling, solvent effects in NMR spectra. 13C NMR – Introduction and theory, Applications. High resolution mass spectral fragmentation mechanism, chemical ionization spectra and extensive analysis of problems based on integrated spectral data with examples chosen from natural products. New dimensions in NMR. Introduction - Theory and applications of DEPT spectra and Homo COSY. Applications of Hetro COSY- HMQC and HSQC. Incredible natural abundance double quantum transfer experiment (2-D INADEQUATE). Problem solving.

(b) Mass spectrometry: Theory, utility of technique, basic principles, isotope abundance, base peak, nitrogen rule, recognition of molecular ion; meta stable ions – calculation of apparent mass – significance –mass spectrometer; fragmentation: general rules for predicting prominent peaks in mass spectra; Mc-Lafferty rearrangement in the case of carbonyl compounds; mass spectra and fragmentation pattern of the following classes of organic compounds: Aliphatic hydrocarbons, aromatic hydrocarbons, alcohols, phenols, ethers, ketones, aldehydes, carboxylic acids, esters, anhydrides, amines, amides, nitriles, nitro compounds, and halides; Illustrations of mass spectral analysis with the help of problems.

(c) Application of UV-VIS, IR, NMR and Mass spectroscopic techniques in the determination of structure of organic compounds (solving combined problems).

Unit 5: Computer aided drug design: Drug design and discovery: an overview, Role of molecular recognition in drug design, Stereochemistry in drug design, Computer-aided development and use of three-dimensional pharmacophore models, Quantitative structure-activity relationships and experimental design, Radiotracer: synthesis and uses in imaging.

References:

1. Organic Spectroscopy, 3rd ed. by William Kemp, Macmillan, 1991.
2. Introduction to Spectroscopy, 2nd ed. by Donald Pavia, Gary M Lamp man, George S Kriz, Saunders college publishing, 1996.
3. Spectrometric Identification of Organic Compounds, 6th ed. by R M Silvenstein, Francis X Webster, John Wiley and sons, 2002.
4. Structural Methods in Inorganic Chemistry, 2nd ed. by E A V Ebsworth, D W H Rankin and S Cradock, Blackwell Publishing, 1991.
5. Principles of Instrumental Analysis, 5th ed. by D A Skoog, F J Holler and T A Nieman, Saunders College Publishing, 1998.
6. Fundamentals of Analytical Chemistry, 5th ed. by D A Skoog, D M West and F U Holler, Saunders, 1988.
7. Textbook of Drug Design and Discovery, 3rd ed. By Povl Krogsgaard-Larsen, Tommy Lilliefors and Ulf Madsen.
8. Principles of Instrumental Analysis, 5th ed. by Douglas A Skoog, James Holler and Timothy A Niemen, Saunders college publishing, 1998.
9. Physical Methods in Inorganic chemistry by R. S. Drago, East-West, 1968.
10. Spectroscopic Techniques for Organic Chemistry by Cooper, Wiley - Interscience, 1980.
11. Contemporary Chemical Analysis by R F Robinson and K A Robinson, Prentice Hall, 1998.
12. Analytical Chemistry, 5th ed. by G D Christian, Wiley. 1986.