**SIDDAGANGA INSTITUTE OF TECHNOLOGY, TUMKUR-572 103.**
**(An Autonomous Institution affiliated to VTU, Belgaum)**

Syllabus from the academic year 2019-20 onwards
**IV Sem. B.E. (Chemical and Biotechnology)**

# INSTRUMENTAL METHODS OF ANALYSIS

| | | | | | |
|---|---|---|---|---|---|
| Contact Hours/Week | : | 3 | Credits | : | 3 |
| Total Lecture Hours | : | 40 | CIE Marks | : | 50 |
| Course Code | : | 4CCB2 | SEE Marks | : | 50 |

**Course Learning Objectives (CLO): (Based on Bloom's Taxonomy)**

**CO1:** To teach students the basic concepts, principles of chromatographic separations and operation of modern chromatographic instrumentation.

**CO2:** To understand the basic principles of electronic spectroscopic techniques and explain the terminology of UV/Vis spectroscopies.

To examine UV spectra based on the knowledge of different electron transitions. .

**CO3:** To know the theory of IR absorption, types of vibrations, factors affecting the group frequencies and sample handling techniques.

To analyze IR spectra based on knowledge of characteristic functional group frequencies.

**CO4:** To demonstrate the knowledge of the chemical shifts and coupling constants in NMR to study $^{1}H$ NMR spectra and propose structures for compounds.

**CO5:** To teach the students about characterization techniques: XRD and electron microscopy which interpret crystal structure and morphology.

**Course Outcomes / Programme Outcomes (POs):**
On successful completion of this course, the graduate will be able to:

- Understand the skills in advanced methods of separation and analysis.
- Study the chromatographic techniques for the identification and purification of compounds.
- Know electronic, IR, mass and NMR spectroscopy in detail.
- Predict electronic spectra, rotational and vibrational spectra for molecules from selection rules.
- Interpret experimental IR, mass and NMR spectra for the identification of unknown organic molecules.
- Study the characterization techniques: XRD and electron microscopy which interpret crystal structure and morphology

**Mapping of Course Outcomes with Program outcomes**

1. Ability to apply knowledge of spectroscopic techniques for qualitative and quantitative analysis.
2. Ability to suggest molecular structure based on experimental spectra.

**Program Articulation Matrix:**

| | POs | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| CO1 | √ | | | | | | | | | | | |
| CO2 | | √ | | | | | | | | | | |
| CO3 | | √ | | | | | | | | | | |
| CO4 | √ | | | | | | | | | | | |
| CO5 | | √ | | | | | | | | | | |

**Course Articulation Matrix:**

| | POs | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| CO1 | 1 | | | | | | | | | | | |
| CO2 | 2 | | | | | | | | | | | |
| CO3 | 2 | | | | | | | | | | | |
| CO4 | 1 | | | | | | | | | | | |
| CO5 | 2 | | | | | | | | | | | |

[1]High association, [2]Moderate association, [3]Low association

## UNIT – I

## CHROMATOGRAPHY

Introduction to Chromatography - Classification - Theory - terminologies- distribution coefficient, retention time, corrected retention time, retention volume, corrected retention volume, retention factor, selectivity factor, column capacity, separation number, peak capacity, column efficiency, resolution and optimization of column performance. Types of chromatography- adsorption, partition, ion exchange and size exclusion chromatography. Numerical problems on retention factor.

### THIN LAYER CHROMATOGRAPHY

Principle, mobile phase, sample application, development techniques – evaluation and documentation, advantages, limitations and applications.

### GAS CHROMATOGRAPHY

Principle, instrumentation, carrier gas, stationary phase, sample injection, columns, detectors (TCD, FID, ECD atomic emission detector). Applications.

## HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Principle, instrumentation, column, sample injection, detectors (UV, refractive index), mobile phase selection, isocratic and solvent gradient system. Applications. **8 Hours**

## UNIT – II

## GENERAL INTRODUCTION TO SPECTROSCOPY

Introduction, Types of spectroscopy-atomic and molecular spectroscopy, nature and interaction of electromagnetic radiations with matter, energies corresponding to various kinds of radiations, spectral band width – definition and factors contributing spectral width, factors influencing positions and intensity of spectral lines. **4 Hours**

## ELECTRONIC SPECTROSCOPY

Principles of electronic spectroscopy - Types of electronic transitions in organic molecules. Chromophores and auxochromes. Bathochromic shift or Red shift, hypsochromic shift or blue shift. Hyperchromic effect and hypochromic effect. Effect of solvent and extent of conjugation on $\lambda_{max}$ and on the energies of n $-\pi^*$ and $\pi - \pi^*$ transitions. Instrumentation, qualitative and quantitative analysis.

**4 Hours**

## UNIT – III

## INFRARED SPECTROSCOPY

Principles of IR spectroscopy. Requirements for IR absorption. Types of vibrations - Stretching vibrations and bending vibrations. Fundamental modes of vibrations for linear and non linear molecules. Characteristic group frequencies for infrared absorption of organic molecules. Factors affecting the group frequencies – coupled interactions (Fermi resonance, aldehyde) electronic effects (carbonyl compounds) and hydrogen bonding (alcohols, carboxylic acids). Numerical problems on vibrational frequencies. Instrumentation-FTIR instrument and its advantages. Sample handling techniques – Nujol mull and KBr pellet. **8 Hours**

## UNIT - IV

## NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

The nuclear spin, Larmor precession, the NMR isotopes, energy levels for a nucleus with spin quantum number I = ½ , $^3/_2$ and $^5/_2$, theory of population of nuclear spin levels, spin-spin and spin-lattice relaxation, chemical shift – definition, causes, measurement. TMS as a reference compound and its advantages, factors affecting chemical shift, shielding and deshielding mechanisms, correlation of chemical shifts with chemical environment – aliphatic, alkenic, alkynic, aldehydic, ketonic, aromatic, alcoholic, phenolic, carboxylic, amino protons, spin – spin

coupling, spin – spin splitting, intensity ratio of multiplet- Pascal's triangle method, chemical exchange, effect of deuteration, classification of spin systems (AX, AMX, AB, ABC), first order spectra, low and high resolution spectra, determination of peak areas, coupling constants-short and long range couplings, Instrumentation – FT NMR. Applications of electronic spectroscopy, IR and NMR to structural elucidation of simple organic molecules. **8 Hours**

**UNIT - V**

**MICROSTRUCTURES AND MORPHALOGICAL STUDIES**

**XRD:** Production of X-rays; types of X-ray sources, Selection of radiation, Braggs Equation, Diffraction by Crystal - direction and intensity of diffracted beams, Calculation of particle size-Debye Scherrer equation proportional, scintillation, solid-state detectors. X-ray spectroscopy for elemental analyses - wavelength dispersive and energy dispersive analyses.

**Microscopy:** Concept of optical microscopy, uses, advantages and disadvantages, Electron microscopy, Introduction, Theory of electron diffraction, Scanning electron microscopy (SEM), AFM – principle and applications, Transmission electron microscopy (TEM), indexing selected area electron diffraction pattern, HRTEM analysis, Comparison of XRD and TEM (HRTEM, SAED pattern) **8 Hours**

**TEXT BOOKS**:


1. Spectrometric Identification of organic compounds, R.M. Silverstein and W.P. Webster, Wiley & Sons, 1999.
2. Instrumental Methods of Analysis. H.H. Willard, L.L. Merritt and J.A. Dean and F. A. Settle, CBS Publishers, 7th Edition, 1988.


**REFERENCE BOOKS**:


1. Instrumental methods of Chemical Analysis, G.W. Ewing, 5th Edition, McGraw-Hill, New York, 1988.
2. Principles of Instrumental Analysis, Skoog, D.A, S.J. Holler, T.A. Nilman, 5th Edn., Saunders college publishing, London, 1998.
3. Instrumental Methods of Chemical Analysis, Chatwal Anand, Himalaya Publishing House.
4. Principles of Electroanalytical Methods, T. Riley and C. Tomilinsom, John Wiley and Sons.
5. Instrumental Methods of Chemical Analysis, K. Sharma, Goel Publishing House, Meerut 2000.
6. Vogel's Text Book of Quantitative Inorganic analysis, Jaffery, Gill, Basset. J 5$^{th}$ Edn., ELBS, 1998.
7. Tools to Characterize Nanomaterials, Textbook of Nanoscience and Nanotechnology, Universities Press (India) Private Ltd 2013


---**---