**Name of the Programme:** M.Sc. Part-I (Chemistry)

**Course Code:** CHA-500 **Title of the course:** Techniques in Analytical Chemistry - I

**Number of Credits:** 04

**Effective from AY:** 2022-23

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| **Prerequisites for the course:** | Students should have studied chemistry courses at graduate level or must have cleared change of discipline entrance test conducted by Goa University. | |
| **Course Objective:** | 1. Learning various methods of data handling in analysis.<br>2. Understanding the significance of sampling and calibration techniques.<br>3. Understanding principles and applications of various types of techniques<br>4. Training the students to deduce structures based on IR, NMR, MS combined data. | |
| **Content:** | **1. Analytical Objectives and Data Handling**<br>Importance of analytical chemistry in research and industry; statistics and data handling in analytical chemistry, standard operating procedures, good laboratory practices: quality assurance, method validation and quality control. | No. of Hours<br>5 |
| | **2. Sampling and Calibration Techniques**<br>Sampling and sample preparation, general steps in chemical analysis, calibration of glass wares. Finding the best straight line-least square regression, correlation coefficient; Calibration curves, standard addition technique and internal standards. Chemical concentrations. | 5 |
| | **3. Classical methods of Analysis**<br>Gravimetry and Titrimetric methods, Principle, methodology, Advantages & Disadvantages over instrumental methods. Conditions for identifying a given reaction as method of Analysis, Classification of reactions in titrimetric analysis (Acid-Base, redox, complexometric and precipitation), Standard solutions and their preparation. Selection of Visual Indicators in titrimetric Analysis | 6 |
| | **4. Introduction to Electroanalytical techniques**<br>Introduction to electrochemical cell, electrode potential, Classification of electroanalytical techniques, working principles, and their applications | 4 |
| | **5. Introduction to Thermoanalytical techniques**<br>Principle, Instrumentation and applications of Thermo Gravimetric Analysis, Differential Thermal Analysis, and Differential Scanning Calorimetry. Numericals based on TGA. | 5 |
| | **6. Introduction to Chromatographic Techniques**<br>a. Principles of chromatography, classification of chromatographic techniques based on mechanism of retention, configuration, mobile and stationary phase. | 15 |

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| | Efficiency of separation- plate theory (theoretical plate concept) and rate theory (van Deemter equation).<br>b. Principles and applications of Paper chromatography, thin layer chromatography, HPTLC, Size exclusion and Ion exchange chromatography. Counter-current chromatography for isolation of natural products.<br>c. Gas and Liquid Chromatography: Introduction; Instrumental Modules; The Separation System; Choice of Conditions of Analysis; Sample Inlet Systems; Detectors; Practical Considerations in Qualitative and Quantitative Analysis; Coupled Systems-introduction to GCMS, LCMS; Applicability-interpretation and numericals. | |
| | **7. Introduction to Spectroscopic Techniques**<br>a. Interaction of Electromagnetic Radiation with Matter: Electromagnetic spectra, regions of spectrum, numericals.<br>b. Ultraviolet and visible Spectroscopy: Electronic spectra and Molecular structure: types of electronic transition, Chromophore and auxochrome, absorption by isolated chromophore, conjugated chromophores, aromatic compounds, inorganic chelates. Calculating λmax for Conjugated Dienes, Trienes, polyenes, α,β-unsaturated carbonyl compounds, Numericals. Choices and effect of solvents on UV-Vis. Quantitative Calculations: Beer-Lambert Law; Mixtures of absorbing species-laws of additivity of absorbance; calibration curve for calculation of unknown; Spectrometric errors in measurement; Deviation from Beer-Lambert Law - chemical deviation, instrumental deviation; Numericals for quantitative analysis using UV-VIS spectroscopy.<br>c. Infrared Spectroscopy: Infrared absorption and molecular structures, molecular vibrations, types of vibrations, IR spectra, overtones and bands-basis of NIR absorption. Spectra interpretation, Frequencies of functional group, Spectral Databases, Identification of unknown compounds.<br>d. Spectrometric Instrumentation of UV-Vis and IR: Sources, monochromators, sample cells, detectors, instrumental wavelength and absorption calibration.<br>e. Proton and Carbon NMR Spectroscopy: Theory of NMR, Instrumentation, Chemical shift, factors influencing chemical shift, solvents used in NMR, spin-spin splitting, coupling constant calculation, factors influencing coupling constant.<br>f. Mass Spectrometry: Principle, Instrumentation and various fragmentation patterns.<br>g. Conjoint spectrometry problems: Structural elucidation of organic molecules using IR, UV, NMR and MS. | 20 |

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| | h. Raman Spectroscopy: Theory, Basic instrumentation and Structural analysis using Raman Spectra.<br>(Note: Assignment based on all above spectrometric methods should be given to student. More weightage of lectures shall be given for solving IR and NMR data problems for structure elucidation) | |
| **Pedagogy:** | Mainly lectures and tutorials. Seminars / term papers /assignments / presentations / self-study or a combination of some of these can also be used. ICT mode should be preferred. Sessions should be interactive in nature to enable peer group learning. | |
| **References / Readings:** | 1. G. D. Christian, Analytical Chemistry, 6th Ed.; Wiley, 2004.<br>2. J. H. Kennedy, Analytical Chemistry: Principles, 2nd Ed.; Saunders College Publishing, 1990.<br>3. G. W. Ewing, Instrumental Methods of Chemical Analysis, 5th Ed.; McGraw- Hill Int., 1985.<br>4. W. Kemp, Organic Spectroscopy, 3rd Ed.; Palgrave, 1991.<br>5. D. A. Skoog, D. M. West, F. J. Hollar, S. R. Crouch, Fundamentals of Analytical Chemistry, 9th Ed.; Cengage learning, 2014.<br>6. F. J. Holler, D. A. Skoog, S. R. Crouch, Principles of Instrumental Analysis, 6th Ed.; Thomson Books, 2007.<br>7. H. Willard, L. L. Merritt, J. A. Dean, F. A. Settle, Instrumental methods of Analysis, 7th Ed.; HCBS Publishing, 2004.<br>8. C. N. Banwell, E. M. McCash, Fundamentals of Molecular Spectroscopy, 4th Ed.; Tata McGraw- Hill, 2006.<br>9. R. M. Silverstein, F. X. Webster, Spectrometric identification of Organic Compounds, 6th Ed.; Wiley, 1998.<br>10. H. Gunzler, A. Williams, Handbook of Analytical Techniques, 1st Ed.; Wiley, 2001.<br>11. P. S. Kalsi, Spectroscopy of Organic Compounds, 2nd Ed.; New Age International, 2000.<br>12. E. Pretsch, P. Buhlmann, C. Affolter, Structural Determination of Organic Compounds, 2nd Ed.; Springer, 2005.<br>13. L. D. Field, S. Sternhell, J. R. Kalman; Organic Structures from Spectra, 4th Ed.; Wiley, 2007.<br>14. R. A. Day, A. L. Underwood, Quantitative Analysis, 6th Ed.; Prentice Hall, 2001.<br>15. B. K Sharma, Instrumental methods of chemical analysis, Goel Publishing House, Meerut, 2004.<br>16. K. Nakamoto, Infrared and Raman Spectra of Inorganic and Coordination Compounds, 6th Ed.; Wiley, 2009.<br>17. P. J. Larkin, Infrared and Raman Spectroscopy: principles and spectral interpretation, 2th Ed.; Elsevier, 2018.<br>18. J. Mendham, R. C. Denney, J. D. Barnes, M. Thomas, B. Sivasankar, Vogel's Text Book of Quantitative Chemical Analysis, 6th Ed.; Pearson, 2009. | |

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| **Course outcomes:** | 1. Students will be able to analyse the role of statistical tools for determination of error and organised data management for systematic interpretation.<br>2. Student will be able to apply the sampling and calibration methods for obtaining reliable results.<br>3. Students will be able to understand basic principles and scope of different methods of Analysis<br>4. Students will be able to solve problems based on IR, NMR, MS combined spectral data. |