Name of the Programme: M.Sc. Part-II (Analytical Chemistry)

Course Code: CHA-622 **Title of the course:** Advanced NMR and combined Spectroscopy

Number of Credits: 4

Effective from AY: 2023-24

Prerequisites for the course:	Students should have studied Chemistry courses in MSc Part-I.	
Course Objective:	1. To understand advance 2D NMR techniques. 2. To develop skills of interpreting spectral data pertaining to two or more 2D NMR techniques. 3. To train students to interpret NMR for quantitative analysis. 4. To understand NMR hyphenated techniques.	
Content	1. **Selected concepts in IR and MS** a. IR: Spectral data interpretation for common functional groups like keto, aldehyde, acid, ester, amides, nitro, etc., Correlation of common functional groups with IR spectral differences. b. MS: Factors governing Mass fragmentation processes, β-cleavage, cleavage α to heteroatoms, cleavage α to carbonyl groups, retro Diels-Alder reaction, McLafferty rearrangement.	No of hours 5
	2. **Selected concepts in NMR** a. Chemical Shifts spectral data for proton and carbon nuclei like aliphatic, aromatic, acyl, methoxy, etc., Correlation of common proton and carbon nuclei with NMR signal differences. b. Nuclear Overhauser Effect c. Decoupling in ^{13}C NMR Spectroscopy (DEPT-45, DEPT-90, DEPT-135), Proton coupled CMR. d. ^{15}N-NMR, ^{19}F-NMR, ^{29}Si-NMR, & ^{31}P-NMR spectroscopy: Chemical shift range for ^{15}N, ^{19}F, ^{29}Si & ^{31}P in NMR spectra, coupling with neighbouring nuclei and splitting pattern.	10
	3. **2D-NMR** a. Introduction to 2D-NMR, General Principles, Classification of 2D-NMR experiments. b. Homonuclear Correlation Spectroscopy Proton-Proton Interactions - COSY, DQF-COSY, TOCSY, NOESY, REOSY. Carbon-Carbon Interactions - INADEQUATE. c. Heteronuclear Correlation Spectroscopy HETCOR Heteronuclear Single Bond Correlation - HSQC, HMQC and me-HSQC Heteronuclear Multiple Bond Correlation - HMBC	10

	d. Analysing and interpreting spectral data from above 2D spectra for small molecules. e. Assigning NMR signals based on PMR, CMR, ^{1}H-^{1}H &^{1}H-^{13}C Correlation Spectra.	
	4. **Structural analysis of simple compounds using some combined spectral techniques**: PMR, CMR, COSY, HSQC, me-HSQC, HMBC, TOCSY, NOESY, INADEQUATE, along with IR, UV and MS data wherever necessary.	20
	5. **Quantitative NMR analysis** a. Analysis of mixture of compounds using qNMR technique, Relative proportions (mole %) of the 2 or 3 components from NMR integrals. b. Calibration standards, Selection criteria for suitable Reference material. c. Molar concentration Determination, Purity or Yield Determination.	10
	6. **Hyphenated NMR techniques** a. Development of LC-NMR, Technical Considerations regarding LC-NMR: Solvent Compatibility, Solvent Suppression, NMR Flow Cell, LC-NMR Sensitivity. Modes of Operation: On-Flow Mode, Stop-Flow Mode. Applications of LC-NMR. b. Introduction to other hyphenated NMR techniques, Technical Considerations regarding LC-MS-NMR: Modes of Operation, Online coupling in series or in parallel, Challenges in Hyphenated NMR techniques.	5
Pedagogy	Mainly lectures and tutorials, Seminars / assignments / presentations / self-study or a combination of some of these can also be used. ICT mode should be preferred. Sessions shall be interactive in nature to enable peer group learning. *(Note: More emphasis shall be given for structural elucidation using combined spectroscopic data*)	
References /Reading	1. W. Kemp; Organic Spectroscopy; 3rd Ed, Palgrave, 1991. 2. R. M. Silverstein, F. X. Webster; Spectrometric identification of Organic Compounds; 6th Ed, Wiley, 2011. 3. R. M. Silverstein, F. X. Webster, D. J. Kiemle, D. L. Bryce, S. D. Samant, V. S. Nadkarni; Spectrometric identification of Organic Compounds; An Indian Adaptation, 8th Ed, Wiley, 2022. 4. P. S. Kalsi; Spectroscopy of Organic Compounds; 6th Ed, New Age International, 2009. 5. E. Pretsch, P. Buhlmann, C. Affolter; Structural Determination of Organic Compounds, 2nd Ed, Springer, 2005. 6. L. D. Field, S. Sternhell, J. R. Kalman; Organic Structures from Spectra, 4th Ed, Wiley, 2007.	

	7. L. D. Field, H. L. Li, A. M. Magill; Organic Structures from 2DNMR Spectra, Wiley, 2015. 8. W. Kemp; NMR in Chemistry: A Multinuclear Introduction, Macmillan, 1986. 9. D. H Williams, I. Fleming; Spectroscopic methods in organic chemistry, 6th Ed, Tata Mcgraw Hill Education, 2011. 10. J. H. Simpson; Organic Structure Determination using 2-D NMR Spectroscopy, Elsevier, 2008. 11. H. Friebolin; Basic One- and Two-Dimensional NMR Spectroscopy, Wiley, 2011. 12. K. S. Parikh, H. H. Gadape; Quantitative NMR Spectroscopy in Pharmaceuticals, Lambert Academic Publishing, 2012. 13. U. Holzgrabe, I. Wawer, B. Diehl; NMR Spectroscopy in Pharmaceutical Analysis, Elsevier, 2008. 14. M. V. Silva Elipe; LC-NMR and Other Hyphenated NMR Techniques: overview and applications, Wiley, 2012.
Course Outcome:	1. Students will be able to understand various 2D NMR techniques and analyse the 2D NMR spectra of small molecules. 2. Students will be skilled to interpret combined spectral data pertaining to two or more 2D NMR techniques for structural analysis. 3. Students will be skilled to interpret qNMR data for quantitative analysis. 4. Students will be able to understand and apply hyphenated NMR techniques for analysing mixtures.