Birla Institute of Technology & Science, Pilani
Hyderabad Campus

First Semester 2022-2023
Course Handout (Part-II)

Date: 29.08.2022

In addition to part I (General Handout for all courses appended to the time table) this portion gives further specific details regarding the course.

Course No.	CHEM F313
Course Title	Instrumental Methods of Analysis
Instructor-in-charge	Ramakrishnan Ganesan
Team of Instructors (Prof./Dr.)	Anupam Bhattacharya, Arijit Mukherjee, Himanshu Aggarwal, Nilanjan Dey, Satya Narayan Guin and Ramakrishnan Ganesan

Course Description:
This course describes the principles and practice of modern instrumental methods of chemical analysis. Emphasis will be given on spectroscopic techniques such as UV-Visible, Infrared, XRD, XPS, XRF, NMR (^1H, ^{13}C and other elements, NOE, correlation spectroscopies), ESR, Mass spectroscopy, atomic absorption and atomic emission spectroscopies, fluorescence spectroscopy and microscopy and chromatographic techniques such as GC/HPLC. Other topics will include electroanalytical methods, thermal analysis and X-ray diffraction methods.

Scope and Objective of the Course:
Chemists extensively use modern sophisticated electronic and optical instruments in various areas such as chemical analysis, structure elucidation, identification of reaction pathways, reaction rates etc. This course aims to introduce the basic theory and experimental details of such instrumentations. Some of the popular absorption spectroscopic techniques like UV-Visible, IR, NMR, etc. will be discussed in detail; other techniques such as XPS, XRD, mass spectrometry, thermal analysis, chromatographic techniques – GC, HPLC, etc. will also be covered.

Text Books:
T1. Lampman G.M., Pavia D.L., Kriz G.S., and Vyvyan J.R., "Spectroscopy", 4th Edition, Cengage Learning (2010).
T2. Gary D. Christian, "Analytical Chemistry", 6th ed., John Wiley & Sons (Asia) Pvt. Ltd. Singapore (2003).

Reference Books:
R1. Kemp W, "Organic Spectroscopy", 3rd ed., Palgrave, New York (1991).

R2. Silverstein R. M., and Webster F. X., “Spectrometric Identification of Organic Compounds”,6th Edition, John Wiley & Sons, New York (1998).
R3. Willard H. H., Merritt L. L., Dean J. A., and Settle F. A. Jr., “Instrumental Methods of Analysis”, 7th Edition. Wadsworth, New York (1989).
R4. Kalsi P. S., “Spectroscopy of Organic Compounds”, 6th Edition, New Age International Publishers, New Delhi (2005).

Course Plan :

A. Lecture Sessions :

Lec. No.	Topics to be covered	Learning Objectives	Learning outcomes	Chapter in the Text Book
1-2	Infrared spectroscopy: Molecular vibrations and chemical bonds; Instrumentation	What is IR spectroscopy? The basic principle and its application to molecules (chemical bonds). The main parts of an IR spectrometer. Sample preparation.	1) Understanding the basis of IR spectroscopy and how Hooke’s law is used in IR spectroscopy. 2) Identify bonds that are IR active. 3) What are the key components/parts in an IR spectrometer? 4) What is FT-IR? 5) How to do sample recording? Solid/Liquid/Gas	2.1-2.6 (T1); class notes
3-4	Applications of Infrared spectroscopy	Factors that influence the vibrational frequency of a bond. Different types of molecules and their IR spectrum	1) Relate IR absorption to factors such as hydrogen bonding, dipole moment, hybridization etc. 2) Insights on the type of bonds by analysing an IR spectrum. 3) Real life applications of IR spectroscopy.	2.7-2.22 (T1); class notes
5-6	Nuclear Magnetic Resonance (NMR) spectroscopy Proton NMR Theory, chemical shift, related factors; NMR instrumentation	Understanding Magnetic Resonance phenomena and the concept of chemical shift; main requirements in NMR spectrometer	1) Identifying magnetically active nuclei. 2) Understanding the importance of nuclear spin. Basis of NMR spectroscopy. 3) Showing the importance of the chemical shift. 4) Types of NMR spectrometers	3.1-3.7 (T1); class notes
7-11	NMR spectra of organic molecules, Solvents, Integrals, spin-spin coupling, related factors, NMR- Nonfirst order spectra, simplification of spectra.	Extracting chemical shift-related structural information from simple NMR spectrum; spin-spin coupling and its effect on the spectrum; What is meant by non-first order NMR spectrum? Different methods of extracting information from such spectra.	1) Solving the structure of a molecule by using NMR data. 2) Type of solvents to be used in NMR. 3) What is spin-spin coupling and its role? 4) Meaning of non-first order spectra and extracting structural information from such spectra.	3.8-3.19 (T1); class notes
12-14	^{13}C NMR technique and its application; NMR for other isotopes ^{19}F, ^{31}P, ^{15}N,	Basis of ^{13}C NMR basis and its interpretation. Interpreting NMR spectra of	1) What is ^{13}C NMR and how to interpret the ^{13}C NMR spectrum? 2) Analysis of compound structure by using ^{1}H NMR and ^{13}C NMR spectrum.	4.1-4.16 (T1); class notes

	^{17}O etc.	nuclei other than ^{1}H and ^{13}C.	3) How to interpret the NMR data for other magnetically active nuclei like ^{19}F, ^{31}P, ^{15}N, ^{17}O etc.?	
15-16	Solid state NMR spectroscopy; Electron Spin Resonance Spectroscopy (Brief discussion)	Basics of solid-state NMR; Principles and applications of electron spin resonance spectroscopy	1) How solid-state NMR is different from NMR for liquid samples ? 2) What is ESR, and how it is useful? 3) Interpretation of the ESR data.	3S.7 (R1); class notes
17-18	Mass spectrometry: Basics, Instrumentation (briefly), Isotopic abundance, and Molecular ion.	Principles of mass spectrometry; parts of a mass spectrometer; the effect of isotopic abundance in the mass spectrum	1) The basic principle of mass spectroscopy. 2) Knowing about parts of a mass spectrometer and their role. 3) Understanding the effect of isotopic abundance in the mass spectrum.	8.1-8.6 (T1)
19-20	Mass spectrometry: fragmentations associated with functional groups	Understanding the molecular fragmentations at the time of ionization and during flight; stabilities of fragments. Extracting the structural information from mass spectra	1) Understanding the molecular fragmentations and stabilities of the fragments generated at the time of ionization and during flight. 2) How to interpret mass spectrum?	8.7 (T1)
21-22	Atomic absorption, emission spectroscopy	Specific atomic energy levels for different elements; instrumentation; quantitative estimations; interferences etc.	1)Will be able to interpret atomic absorption spectra 2) Explain the basic principles of AAS. 3)Can Illustrate the working principle and outline of AAS 4) Recall Maxwell's distribution law 5) Discuss the above similarities with Flame emission spectrophotometry	Ch. 17 (T2)
23	Energy and Electromagnetic spectrum	Regions of Electromagnetic Spectrum; units.	1) Explain the interaction between light and matter 2) Contrast various regions of the electromagnetic spectrum 3) Estimate the energy of transition and relate to the units	Ch.1 (R1); 16.1 (T2)
24-27	Ultraviolet (UV) and visible spectroscopy: Light Absorption, theory, instrumentation	Chromophore concept; electronic energy levels.	1)Relate the basic principle of UV-Vis spectroscopy and explain relevant terms 2) Outline the working principle, analyzing the spectra and extend the construction of device	4.1-4.3 (R1);16.2 (T2)
28-29	UV-Visible: Solvents, applications	Solvent effects; Absorption wavelength calculations based on empirical rules	1)Recall the basic concepts of electronic transitions and organize the study of solvent effect on UV-Spectra 2)Calculate the wavelength of absorption in conjuagted systems using Woodward rule	4.4-4.10 (R1)
30-32	Fluorescence and phosphorescence	Principles of fluorescence and phosphorescence and applications	1)Define fluorescence and phosphorescence 2)Elaborate Jablonskii diagram 3)Interpret fluorescence property of the molecules 4)Decide quenching phenomenon 5)Fluorescence lifetime and its applications 6) Fluorescence microscopy	4S.2 (R1) & 16.15 (T2)
33-34	Thermo analytical methods	Differential Thermal Analysis; Thermo	1)Define and demonstrate the thermoanalytical methods: DTA, TGA and DSC	Ch. 20 (R3)

		Gravimetric Analysis; Differential Scanning Calorimetry etc.	2)Conclude the changes in the sample, exothermic or endothermic can be detected relative to the inert reference 3) Develop knowledge pertaining to the appropriate use of the instrument for thermal analysis.	
35-36	Chromatographic Techniques: GC, HPLC, Electrophoresis	Theories of separation techniques; stationary and mobile phases etc.	1)Infer the theoretical aspects of techniques used for separation 2) Make use of mobile and stationary phases and estimate certain physical parameters dealing with the above-mentioned techniques	Ch. 19, 20.1-20.3, 21.1-21.3; 21.5(T2)
37-40	Characterization of materials by XRD, XRF, Auger spectroscopy and XPS	Basic theory and applications in characterizing various materials	Understanding the basis X-ray based absorption and emission techniques	Lecture notes

B. Practical Sessions:

Regular sessions: (10 to 12 sessions)

In these sessions, the students (in groups) will perform an experiment on various instrumental techniques such as UV-Visible spectroscopy, spectrofluorimetry, IR spectroscopy, AAS, TGA, DSC, NMR, CD, Mass spectrometry, etc. All students are required to write a report about the performed experiment and submit it during the next session. The instructors will make procedure sheets available for each of these laboratory experiments.

Evaluation Scheme: **Total 200 marks**

A. Theory (150 Marks/75% Weightage)

Components	Duration	Weightage	Date & Time	Nature of Component
Mid-Sem	1.5 h	25 %	01/11 9.00 - 10.30AM	Closed Book
Class tests*	---	10 %	Continuous	Closed Book
Comprehensive Examination**	3 h	40 %	20/12 FN	Closed Book

* There will be 4 class tests of each 5 marks. All the four are mandatory.

** The mid-semester and comprehensive examination can have objective and descriptive portions.

B. Practical (50 Marks/25% Weightage – Open book)

There will be **ten regular experiments**: Each experiment shall carry 5 marks out of which 3 would be for record submission and 2 would come from a quiz.

Chamber consultation hour: Anupam Bhattacharya (TBA); Ramakrishnan Ganesan (Thursday from 12.00 noon to 1.00pm)

Makeup Policy: See Part I for details. However, it may be noted that it is difficult to arrange the make-up of practical sessions.

Notices: All the notices pertaining to this course will be displayed on the **CMS**.

Academic Honesty and Integrity Policy: Academic honesty and integrity are to be maintained by all the students throughout the semester, and any type of academic dishonesty is unacceptable.

Course Policies:

Absences: Students are responsible for all the materials presented in the course as well as for acquiring missed information.

Ramakrishnan Ganesan
Instructor-in-charge
CHEM F313