**BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI – Hyderabad Campus**

**AUGSD**

**First Semester 2021-2022**

**Course Handout**

Date: 18.08.2021

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

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| **Course No.** | CHEM G554 |
| **Course Title** | Physical Methods in Chemistry |
| **Instructor-in-charge** | Nilanjan Dey |
| **Team of Instructors** | Anupam Bhattacharya, Arijit Mukherjee |

**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, XPS, XRF, NMR (1H, 13C and other elements, NOE, correlation spectroscopies), ESR, Mass spectroscopy, atomic absorption and emission spectroscopies, fluorescence spectroscopy and microscopy and chromatographic techniques such as GC/HPLC. Other topics will include electroanalytical methods, thermal analysis and diffraction methods like XRD.

**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 such as 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. Kemp W, “Organic Spectroscopy”, 3rd ed., Palgrave, New York (1991).

T2. Lampman G.M., Pavia D.L., Kriz G.S., and Vyvyan J.R., “Spectroscopy”, 4th Edition, Cengage Learning (2010).

T3. W. Massa, Crystal Structure Determination ], 2nd English Edition, Springer-Verlag Berlin Heidelberg (2004)

**Reference Books:**

R1. Silverstein R. M., and Webster F. X., “Spectrometric Identification of Organic Compounds”,6th Edition, John Wiley & Sons, New York (1998).

R2. Willard H. H., Merritt L. L., Dean J. A., and Settle F. A. Jr., “Instrumental Methods of Analysis”, 7th Edition. Wadsworth, New York (1989).

R3. Kalsi P. S., “Spectroscopy of Organic Compounds”, 6th Edition, New Age International Publishers, New Delhi (2005).

**Course Plan :**

**A. Lecture Sessions :**

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| **Lec. No.** | **Topics to be covered** | **Learning Objectives** | **Learning outcomes** | **Reference:**  **Chap./Sec.**  **#(Book)** |
| 1 | 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   Estimate the energy of transition and relate to the units | Ch.1 (T2) |
| 2 | 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 (T2) |
| 3-4 | 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 (T2) |
| 5 | Circular Dichroism Spectroscopy | Principle of Circular Dichroism, details and applications | 1) Principle and basics of CD and ORD spectrosocpy  2) Analyzing an CD spectrum, information about the chirality  3) Application of CD spectroscopy in biomolecular analysis (DNA and protein) | Lecture Notes |
| 6-7 | 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 (T1) &  16.15 (T2) |
| 8 | Infrared spectroscopy: Molecular vibrations; related factors | IR absorption due to molecular vibrations; influence of factors such as hydrogen bonding. | 1) Understanding the basis of IR spectroscopy and how Hooke’s law is  used in IR spectroscopy.  2) Identify bonds which are IR active.  3) Relate IR absorption to factors such as hydrogen bonding, dipole  moment, hybridization etc. | 2.1-2.3 (T2) |
| 9 | Infrared spectroscopy: Instrumentation, Applications | IR instrumentation details; FT-IR; sample preparations recording details | 1. What are the key components/parts in an IR spectrometer? 2. What is FT-IR? 3. How to do sample recording? Solid/Liquid/Gas   Basis of using a particular compound for sample preparation. | 2.4-2.7 (T1) &  2.1-2.9 (R1) |
| 10-11 | Infrared spectroscopy: Correlation charts; Supplementary materials | Obtaining structural information from IR spectrum; Reflectance mode IR spectra | 1. Analysis of an IR spectrum, to obtain information about presence of functional groups and also examine the possibility of getting some structural insights. 2. IR in the reflectance mode; key aspects. | 2.8-2S.3 (T1) &  2.10-2.21 (R1) |
| 12-13 | Raman Spectroscopy | Principles of Raman spectroscopy and applications | 1) Understanding the basics of Raman spectroscopy  2) Key differences with IR  3) Analysis of an Raman spectrum, to obtain information about the functional groups and structural insights. | Lecture Notes |
| 14-15 | 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) |
| 16-17 | Thermo analytical methods | Differential Thermal Analysis; Thermo Gravimetric Analysis; Differential Scanning Calorimetry etc. | 1)Define and demonstrate the thermoanalytical methods: DTA, TGA and DSC  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. | Ch. 20 (R3) |
| 18-19 | Characterization of materials by XPS and XRF | Basic theory and applications in characterizing various materials | Understanding the basis X-ray based absorption and emission techniques | Lecture notes |
| 20-24 | Characterization of materials by XRD | Basic theory of XRD and its application in characterizing different inorganic material | 1. Define lattice, crystal systems  2. Explain the mechanism of X-ray Generation  3. Explain diffraction with Laue and Bragg equations  4. Define Ewald Construction to depict reciprocal space  5. Define and use of symmetry elements  6. Analysis of powder diffractograms in a qualitative manner | Lecture notes |
| 25-26 | 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, 21.1(T2) |
| 27-28 | Nuclear Magnetic Resonance (NMR) spectroscopy Proton NMR Theory, chemical shift, related factors | Understanding Magnetic Resonance phenomena and the concept of chemical shift | 1. Identifying magnetically active nuclei. 2. Understanding the importance of nuclear spin. Basis of NMR spectroscopy.   Showing the importance of the chemical shift. | 3.1-3.4 (T1) |
| 29-31 | NMR- Correlation Data, Solvents, Integrals, spin-spin coupling, related factors | Extracting chemical shift related structural information from simple NMR spectrum; spin-spin coupling and its effect on the spectrum | 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? | 3.5-3.9 (T1) |
| 32-33 | NMR- Non first order spectra, simplification of spectra, tables, 13C NMR applications | What is meant by non-first order NMR spectrum; different methods of extracting information from such spectra; 13C NMR how to interpret. | 1) Meaning of non-first order spectra and extracting structural information from such spectra.  2) What is 13C NMR and how to interpret 13C NMR spectrum? | 3.10-3.16 (T1) |
| 34-35 | NMR-double irradiation, multi pulses, NMR for other isotopes 19F, 31P, 15N, 17O etc. | Understanding a few of the advanced methods in NMR; Interpreting NMR spectra of nuclei other than 1H and 13C | 1. What are the various advanced methods in NMR and how to obtain molecular structure-related information from them? 2. How to interpret the NMR data for other magnetically active nuclei like 19F, 31P, 15N, 17O etc.? | 3S.1-3S.6 (T1) |
| 36-37 | Mass spectrometry: Basics, Instrumentation, Isotopic abundance, and Molecular ion. | Principles of mass spectrometry; the effect of isotopic abundance in the mass spectrum | 1. The basic principle of mass spectroscopy. 2. Understanding the effect of isotopic abundance in the mass spectrum. | 5.1-5.4 (T1) &  8.3-8.5 (R1) |
| 38-39 | Mass spectrometry: Metastable ions, fragmentation processes | Understanding the molecular fragmentations at the time of ionization and during flight; stabilities of fragments. | 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? | 5.5-5.6 (T1) |

Evaluation Scheme: Total 200 marks

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| **Components** | **Duration** | **Weightage** | **Date & Time** | **Remarks** |
| Mid-Sem | --- | 26 % | --- | Open Book |
| Seminar | --- | 39 % | Continuous | Open Book |
| Comprehensive Examination\*\* | --- | 35 % | --- | Open Book |

\* There will be 3 seminars, each carrying 13 % weightage

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

**Makeup Policy:** See Part I for details. However, it may be noted that it is difficult to arrange to 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 no type of academic dishonesty is acceptable.

**Course Policies:**

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

Nilanjan Dey

Instructor-in-charge

CHEM G554