

BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI – Hyderabad Campus
INSTRUCTION DIVISION
First Semester 2018-2019
Course Handout (Part-II)

Date: 20.07.2019

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 : Amit Nag, Anupam Bhattacharya, N. Rajesh, Sounak Roy 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, XPS, XRF, NMR (^1H , ^{13}C 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. Gary D. Christian, “Analytical Chemistry”, 6th ed., John Wiley & Sons (Asia) Pvt. Ltd. Singapore (2003).
T2. Kemp W, “Organic Spectroscopy”, 3rd ed., Palgrave, New York (1991).

Reference Books:

- R1. Lampman G.M., Pavia D.L., Kriz G.S., and Vyvyan J.R., “Spectroscopy”, 4th Edition, Cengage Learning (2010).

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 | Reference: Chap./Sec. # (Book) |
|----------|--|---|--|--------------------------------|
| 1-2 | Atomic absorption, emission spectroscopy | Specific atomic energy levels for different elements; instrumentation; quantitative estimations; interferences etc. | 1) Will be able to interpret atomic absorption spectroscopy 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 (T1) |
| 3-4 | 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 (T1) |
| 5 | 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) |
| 6 | Electro analytical methods | Analytical methods based on measurements of current voltage etc. | 1) State the basic principles under electroanalytical techniques 2) Mention and explain various methods for the determination of physical parameters coming across these techniques 3) Explain the concepts of electroanalytical and electrochemical cells 4) Extend basic equations of electrochemistry and their applications to electro analysis | Ch. 15 (T1) |
| 7 | 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 (T2) |
| 8 | 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 3) Evaluate absorption parameters | 4.1-4.3 (T2) |

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| 9-10 | 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 conjugated systems using Woodward rule | 4.4-4.10 (T2) |
| 11-12 | 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 (T2) & 16.15 (T1) |
| 13 | 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) |
| 14 | 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? <u>Solid/Liquid/Gas</u> 4) Basis of using a particular compound for sample preparation. | 2.4-2.7 (T2) & 2.1-2.9 (R1) |
| 15 | 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 reflectance mode ; key aspects. | 2.8-2S.3 (T2) & 2.10-2.21 (R1) |
| 16-18 | 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 |
| 19 | Characterization of materials by XRD | Basic theory of XRD and its application in characterizing different inorganic material | Analyzing ability of diffractograms from the XRD study | Lecture notes |
| 20-21 | 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. 3) Showing the importance of chemical shift. | 3.1-3.4 (T2) |
| 22-25 | 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 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 (T2) |
| 26-29 | NMR- Non first order | What is meant by non-first | 1) Meaning of non-first order spectra and extracting structural | 3.10-3.16 (T2) |

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|-------|--|--|---|-----------------------------|
| | spectra, simplification of spectra, tables, ^{13}C NMR applications | order NMR spectrum; different methods of extracting information from such spectra; ^{13}C NMR how to interpret. | information from such spectra. 2) What is ^{13}C NMR and how to interpret ^{13}C NMR spectrum? | |
| 30-32 | NMR- double irradiation, multi pulses, MRI, polarization techniques, other isotopes ^{19}F , ^{31}P , ^{15}N , ^{17}O etc. | Understanding a few of the advanced methods in NMR; Interpreting NMR spectra of nuclei other than ^1H and ^{13}C | 1) What are the various advanced methods in NMR and how to obtain molecular structure related information from them ? 2) How to interpret NMR data for other magnetically active nuclei like ^{19}F , ^{31}P , ^{15}N , ^{17}O etc. ? 3) What is the basis of MRI ? | 3S.1-3S.6 (T2) |
| 33-34 | Electron Spin Resonance Spectroscopy | Principles and applications of electron spin resonance spectroscopy | 1) What is ESR and how it is useful ? 2) Interpretation of ESR data. | 3S.7 (T2) |
| 35-36 | Mass spectrometry: Basics, Instrumentation, Isotopic abundance, and Molecular ion. | Principles of mass spectrometry; the effect of isotopic abundance in the mass spectrum | 1) Basic principle of mass spectroscopy. 2) Understanding the effect of isotopic abundance in the mass spectrum. | 5.1-5.4 (T2) & 8.3-8.5 (R1) |
| 37-38 | 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. | 5.5-5.6 (T2) |
| 39-41 | Mass spectrometry: fragmentations associated with functional groups | Extracting the structural information from mass spectra | 1) How to interpret mass spectrum? | 5.7 (T2) & 8.6 (R1) |
| 42 | Mass spectrometry: Supplementary topics | Understanding different kind of mass spectrometers, hyphenated techniques such as GC-MS, isotopic substitution etc. | 1) What are the different kind of mass spectrometers and the techniques they use ? | 5S.1-5S.5 (T2) & 8.2 (R1) |

B. Practical Sessions:

Regular sessions: (10 to 12 sessions)

In these sessions the students (in groups) will perform an experiment using the techniques such as UV-Visible spectroscopy, spectrofluorimetry, IR spectroscopy, AAS, TGA, DSC, GC/HPLC, NMR, ED-XRF, XRD and XPS. 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. There will be two demo sessions on two more techniques namely FE-SEM and LC-MS.

Evaluation Scheme: Total 300 marks

A. Theory (195 Marks/65% Weightage)

| Components | Duration | Weightage | Date & Time | Remarks |
|-----------------------------|----------|-----------|-------------------------|---------|
| Mid-Sem | 1.5 h | 20% | 3/10, 11.00 -- 12.30 PM | CB |
| Surprise tests* | --- | 10% | Continuous | CB |
| Comprehensive** Examination | 3 h | 35% | 09/12 AN | CB |

* There will be 6 surprise tests, each carrying 6 marks. Best 5 of them will be taken for final evaluation. ** The comprehensive examination will have objective and descriptive portions.

B. Practical (105 Marks/35% Weightage OB)

There will be **ten regular experiments and two demonstration sessions**: Each experiment shall carry 8 marks (Practical 4; Records 4) to a total of 80 marks. A pen & paper Quiz (closed book) related to the experiments, will be performed on a later date and will carry 25 marks.

Chamber Consultation Hours: Will be announced later.

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

Notices: Notices, if any, concerning the course will be displayed on the notice board of Chemistry Department only.

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.

Ramakrishnan Ganesan
Instructor-in-charge
CHEM F313