

BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI – Hyderabad Campus
INSTRUCTION DIVISION
First Semester 2016-2017
Course Handout (Part-II)

Date: 21.07.2016

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: Ramakrishnan Ganesan, Manab Chakravarty, N. Rajesh, Balaji Gopalan, R. Krishnan.

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, NMR (^1H , ^{13}C and other elements, NOE, correlation spectroscopies), ESR, Mass spectroscopy, atomic absorption and emission spectroscopies, fluorescence and chromatographic techniques such as GC/ HPLC. Other topics will include separation techniques, light scattering, electroanalytical methods, thermal analysis, and diffraction methods.

Scope and Objective of the Course:

Chemists extensively use modern sophisticated electronic 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 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. Gary D. Christian, “Analytical Chemistry”, 6th ed., John Wiley & Sons (Asia) Pvt. Ltd. Singapore (2003).

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	Reference: Chap./Sec.#(Book)
1	Infrared spectroscopy: Molecular vibrations; related factors	IR absorption due to molecular vibrations; influence of factors such as hydrogen bonding.	2.1-2.3 (T1)
2-3	Infrared spectroscopy: Instrumentation, Applications	IR instrumentation details; FT-IR; sample preparations recording details	2.4-2.7 (T1) & 2.1-2.9 (R1)
4-6	Infrared spectroscopy: Correlation charts; Supplementary materials	Obtaining structural information from IR spectrum; Reflectance and Raman spectroscopies comparison	2.8-2S.3 (T1) & 2.10-2.21 (R1)
7-8	Nuclear Magnetic Resonance (NMR) spectroscopy Proton NMR Theory, chemical shift, related factors	Understanding Magnetic Resonance phenomena and the concept of chemical shift	3.1-3.4 (T1)
9-12	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	3.5-3.9 (T1)
13-16	NMR- Non first order spectra, simplification of spectra, tables, ^{13}C NMR applications	What is meant by non-first order NMR spectrum; different methods of extracting information from such spectra; ^{13}C NMR how to interpret.	3.10-3.16 (T1)
17-19	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	3S.1-3S.6 (T1)
20-21	Electron Spin Resonance Spectroscopy	Principles and applications of electron spin resonance spectroscopy	3S.7 (T1)
22	Atomic absorption, emission spectroscopy	Specific atomic energy levels for different elements; instrumentation; quantitative estimations; interferences etc.	Ch 17 (T2)
23-25	Chromatographic Techniques: GC, HPLC, Electrophoresis	Theories of separation techniques; stationary and mobile phases etc.	Ch. 19 20.1, 21.1(T2)
26-27	Thermo analytical methods	Differential Thermal Analysis; Thermo Gravimetric Analysis; Differential Scanning Calorimetry etc.	Ch. 20 (R3)
28	Electro analytical methods	Analytical methods based on measurements of current voltage etc.	Ch. 15 (T2)

29	Energy and Electromagnetic spectrum	Regions of Electromagnetic Spectrum; units.	Ch.1 (T1)
30	Ultraviolet (UV) and visible spectroscopy: Light Absorption, theory, instrumentation	Chromophore concept; electronic energy levels.	4.1-4.3 (T1)
31-33	UV-Visible: Solvents, applications	Solvent effects; Absorption wavelength calculations based on empirical rules	4.4-4.10 (T1)
34	Fluorescence and phosphorescence	Principles of fluorescence and phosphorescence and applications	4S.2 (T1) & 16.15 (T2)
35	Mass spectrometry: Basics, Instrumentation, Isotopic abundance, and Molecular ion.	Principles of mass spectrometry; the effect of isotopic abundance in the mass spectrum	5.1-5.4 (T1) & 8.3-8.5 (R1)
36-37	Mass spectrometry: Metastable ions, fragmentation processes	Understanding the molecular fragmentations at the time of ionization and during flight; stabilities of fragments.	5.5-5.6 (T1)
38-40	Mass spectrometry: fragmentations associated with functional groups	Extracting the structural information from mass spectra	5.7 (T1) & 8.6 (R1)
41-42	Mass spectrometry: Supplementary topics	Understanding different kind of mass spectrometers, hyphenated techniques such as GC-MS, isotopic substitution etc.	5S.1-5S.5 (T1) & 8.2 (R1)

B. Practical Sessions:

Regular sessions: (10 to 12 sessions)

In these sessions the student (in groups) will perform a simple experiment in the techniques such as UV-Visible spectroscopy, spectrofluorimetry, IR spectroscopy, polarimetry, AAS, HPLC/GC analysis, flame photometry, DSC, X-ray fluorescence etc. All students are required to write a report and come prepared for viva-voce examination during next session. The instructors will make procedure sheets available for each of these laboratory experiments.

Evaluation Scheme: Total 300 marks

A. Lecture (180 Marks)

Components	Duration	Marks	Date & Time	Remarks
Test I	1 h	40	---	CB
Test II	1 h	40	---	CB
Surprise tests (Best 4 out of 6)	---	20	Continuous	CB
Comprehensive* Examination	3 h	80	---	CB

* The comprehensive examination will have objective and descriptive portions.

B. Practical (120 Marks)

For the ten regular sessions: 10 marks each per session (Practical 5; Records 5). Quiz will carry 20 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.

Ramakrishnan Ganesan
Instructor-in-charge
CHEM F313