BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI
INSTRUCTION DIVISION
First Semester 2015-2016
Course Handout (Part-II)

Date: 03/08/2015

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 **:** BIBHAS R. SARKAR

Team of Instructors : **:** Ram K. Roy, Saumi Ray, Indresh Kumar

Course Description: Principles and practice of instrumental methods of chemical analysis and different chemistry applications will be studied. A wide range of analytical techniques used in chemical applications will be introduced with respect to their principles, instrumentation, applications etc. Emphasis will be given on several spectroscopic techniques such as but not limited to, UV-Visible, FT-IR, NMR (^{1}H & ^{13}C), mass spectrometry, atomic absorption and emission, Microscopy (electron and optical) techniques, thermal methods, fluorescence, and Mössbauer etc.

Scope and Objective of the Course: Chemists extensively use modern sophisticated electronic instruments in various areas such as chemical analysis, structure determination, identification of reaction pathways and rates etc. This course aims to introduce the basic theory and experimental details of such chemical instrumentation. Some of the popular absorption spectroscopic techniques such as UV-visible, IR, NMR, mass spectrometry etc. will be discussed in detail.

Text Books: T1. Kemp W., "Organic Spectroscopy", 3rd ed., Palgrave, New York (1991).

Reference Books:

R1. Willard H. H., Merritt Jr. L. L., Dean J. A., Settle F. A. S., "Instrumental Methods of Analysis", 7th Ed., Wadsworth, 2009, Cengage Learning India Pvt. Ltd. Fifth Indian reprint by CBS Publishers & Distributors Pvt. Ltd.

R2. Silverstein R. M., and Webster F. X., "Spectrometric Identification of Organic Compounds", 6th ed., John Wiley & Sons, New York (1998).

R3. Skoog D. A., Holler F. J., and Crouch S. R., "Principles of Instrumental Analysis", 6th ed., Thomson Brooks/Cole, Cengage Learning, New Delhi (2007).

Course Plan: A. Lecture Sessions:

Lec. No.	Topics to be covered	Learning Objectives	Reference: Chap./Sec.#(Book)
1	Introduction to Chemical analysis, Instrumental methods Measurements, Signals and Data	Classification of techniques, and their application windows Understanding of essential components of instrument use, Errors, accuracy, calibration methods	Ch 1-2 (R1)
2	Introduction to Energy and Electromagnetic spectrum, Absorption and emission spectroscopy.	Regions of Electromagnetic Spectrum; units. Correlation and outline of the course	Ch.1 (T1) Ch 5 (R1)

3	Ultraviolet (UV) and visible spectroscopy: Light Absorption, theory, instrumentation	Chromophore concept; electronic energy levels; differences in dispersive and other instruments.	Ch. 4.1-4.3(T1)
4	UV-visible: solvents, applications	Solvent effects; Absorption wavelength calculations based on empirical rules.	Ch. 4.4-4.10(T1)
5-6	UV-visible: stereo chemical factors, quantitative electronic spectroscopy	Stereo chemical factors which change the conjugation; quantitative estimation calculations	Ch. 4.11-4S.1(T1) Ch 6-7 (R1)
7	Fluorescence and phosphorescence	Principles of fluorescence and phosphorescence and applications	Ch. 4S.2 (T1)& Ch. 8 (R1)
8	UV-visible: CT complexes, symmetry, ORD-CD	Principles, instrumentation and some simple applications of ORD, CD,	Ch. 4S.3-4S.6(T1)
9	Infrared spectroscopy: Molecular vibrations; related factors	IR absorption due to molecular vibrations; influence of factors such as hydrogen bonding.	Ch. 2.1-2.3(T1)
10-12	Infrared spectroscopy: Instrumentation, and Applications	IR instrumentation details; FT-IR; sample preparations recording details, different modes, structural elicidation using FT-IR	Ch. 2.4-2.8 (T1) Ch 11 (R1)
13	Raman spectroscopy	Obtaining structural information from IR spectrum; Reflectance and Raman spectroscopies comparison	Ch.2S.3(T1) Ch 12 (R1)
14-20	Nuclear Magnetic Resonance (NMR) spectroscopy	Understanding Magnetic Resonance phenomena and the concept of chemical shift, structural information from simple NMR spectra; spin-spin coupling and its effect on the spectrum, interpretation of ^{1}H NMR, ^{13}C NMR spectra, Quantitative Analysis, Applications	Ch 3 (T1) Ch 15 (R1)
21-22	Electronic Paramagnetic Resonance (EPR)	Basic principle, instrumentation	Class notes
23-26	Mass spectrometry: Basics, Instrumentation, Isotopic abundance, and Molecular ion. metastable ions, fragmentation processes	Principles of mass spectrometry; the effect of isotopic abundance in the mass spectrum, Understanding the molecular fragmentations,; stabilities of fragments. Structure elucidation	Ch 5 (T1)
27-28	Flame Emission & Atomic Absorption Spectroscopy	Introduction to flame photometry, atomic absorption, fluorescence, applications	Ch 9 (R1)
29	Atomic emission Spectroscopy (with plasma and electrical discharge)	ICP-methods, applications	Ch 10 (R1)
30-32	X-ray Methods	Understand the Basics, X-ray absorption, X-ray fluorescence, X-ray Diffractions, Auger Spectroscopy	Ch 13 (R1) Ch. 8-9(R3), Lecture notes
33-34	Microscopy Techniques	Optical and Electronic microscopy including, SEM, STEM, TEM etc	Lecture notes

35	Thermal Analysis	Differential scanning calorimetry, Thermogravimetry, Evolved gas detection & analysis	Ch. 25(R1) Ch. 31 (R3)
36	Radiochemical/ Nuclear Methods	Radiations, Nuclear reactions, Neutron activation analysis, other techniques	Ch 14 (R1)
37	Electroanalytical methods	Introduction and brief descriptions on potentiometry, voltametry etc.	Ch 8 (11-24)
38-39	Separation Techniques: Chromatography – Basics and applications,	Classification, behavior of solutes, Quantitative analyses, Theories of different techniques such as HPLC, GC etc.	Ch 17-20 (R1), Ch 27-30 (R3)
40	Hyphenated techniques, Applications	Understanding different kind of mass spectrometers, hyphenated techniques such as GC-MS, MS-MS, ICP-MS, isotopic substitution etc	Ch. 5S.1-5S.5(T1)

B. Practical Sessions:

Regular sessions: (8-10 sessions)

In these sessions the student (in groups) will perform a simple experiment in the techniques such as UV-visible spectroscopy, spectrofluorimetry, IR spectroscopy, , flame photometry, HPLC analysis, High voltage electrophoresis, polarimetry, DSC analysis. Demonstration few more instrumental techniques will also be done including NMR spectroscopy, GC analysis, Cyclic Voltammetry, etc. The instructors will make available experimental handout sheets for each of these laboratory experiments.

All students are required to

1. Write a report of the experiment in the next session
2. Come prepared for viva-voce examination during the experiments respectively.

Project sessions: (1 or 2 sessions)

The students will form groups (not more than four students in a group) and select a project amongst the titles provided by the instructors. The project may involve more than one technique learnt in the regular sessions. Students can also suggest projects; however, the instructor's approval is necessary. Projects involving electronics/instrumentation will also be accepted.

USE OF APPROPRIATE PERSONAL PROTECTIVE EQUIPMENT (PPE) (e.g. Apron, gloves, covered footwear etc.) IS MANDATORY DURING ALL LABORATORY SESSIONS. DEFAULTERS WILL NOT BE ALLOWED TO PERFORM WITHOUT PPE.

Evaluation Scheme:

Total marks: 300

A. Lecture (210 Marks)

Components	Duration	Marks	Date & Time	Venue	Remarks
Mid-semester test	90 min	60	10/10 2:00 - 3:30 PM		
Tutorial tests	20 min	50	Tutorial Class		@
Comprehensive Examination	3 hours	100	14/12 FN		$

@ **Tutorial:** The tutorial hour will be used for a quick review of the highlights of the material covered in the lectures, clarification of doubts and problem solving. Further, a set of problems will be assigned periodically, of which the Instructor will specify some to be solved by the students in the tutorial hour of the following week.
The second method of continuous evaluation in tutorial will be of a Tutorial test/ short quiz based on the lectures covered recently. Each tutorial evaluation will be for ten marks. Total 6 evaluation components in tutorial, with best 5 performances to be counted.

$ The comprehensive examination will have a closed book portion for 20 marks, and an open book section for 80 marks.

B. Practical (90 Marks)
Nine marks each for the nine regular sessions and nine marks for the project. If there are more than ten experiments (including the project) then best ten experiments will be taken into account.

Chamber Consultation Hours: To be announced

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

Notices: Notices, if any, concerning the course will be displayed on the Nalanda and/or Department of Chemistry Notice board

Instructor-in-charge
CHEM F313