



**Birla Institute of Technology & Science, Pilani**  
Hyderabad Campus

**Second Semester 2018-2019**

Course Handout Part II

Date: 07-01-2019

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.* : **PHA F313**  
*Course Title* : **Instrumental Methods of Analysis**  
*Instructor-in-Charge* : **A. SAJELI BEGUM**  
*Instructors* : Pragya, Shubham, Himanshu and Deepanjan

### **Scope and Objective of the Course:**

The course is aimed at exposing the students to modern analytical techniques in relevance to pharmaceutical industries. Training with respect to the handling of sophisticated instruments and their operation, interpretation of results obtained with relevance to the identification and characterization of molecules, their qualitative and quantitative evaluation and control are emphasized.

### **Learning Outcome:**

- Handling of analytical instruments for performing qualitative and quantitative analyses of drugs.
- Design and development of suitable analytical methods for drugs based on their physico-chemical characters meeting the standard requirements.
- Ability to interpret the analytical graphs and spectra for drug identification, characterization, evaluation and decision making

### **Textbooks:**

- T1. Willard H.H., et al., "Instrumental Methods of Analysis", CBS Pub., New Delhi, 7<sup>th</sup> ed., 1988  
T2. Parimoo P, "Pharmaceutical Analysis", CBSI, 1988

### **Reference books**

- R1. Robert M Silverstein, Francis X Webster -"Spectroscopic Identification of Organic Compounds", Eighth Edition, John Wiley and Sons, Inc., N.Y., 2014.  
R2. A.H. Beckett and J.B. Stenlake -"Practical Pharmaceutical Analysis", Fourth Edition, CBS Pub., New Delhi., Vol. I & II, 1988.  
R3. Maureen Melvin,-"Electrophoresis", John Wiley and Sons, Inc., N.Y., 1987.  
R4. B.G.Nagavi,-"Laboratory Handbook of Instrumental Drug Analysis", Vallabh Prakashan, New Delhi, 1996.  
R5. Mahesh, R & Others Instru. Methods of Analysis Lab Manual Notes EDD, 2007.



**Course Plan:****a. Lecture Plan**

Lect No.	Learning Objectives	Topics	Chapter in the Text Book
1	Introduction of various analytical techniques applicable in field of Pharmacy	Introduction to Instrumental Methods of Analysis	TB-1: 1-5; TB-2: 1,2
2-6	Learn the principle, instrumentation and application - Difference, Derivative Spectroscopy	UV Spectroscopy	TB-1: 6, 7; TB-2: 14,15 TB-1: 2,3; TB-2:14,15 and R4
7-11	Learn the theory, instrumentation, spectral data analysis followed by applications in preformulation studies	IR Spectroscopy	TB-1: 11 ; TB-2: 18; R2-3
12-13	Theory and factors governing spectrofluorimetry	Spectrofluorimetry	TB-1: 8; TB-2: 17
14	Theory and applications	Polarimetry	TB-2: 10
15-23	Knowledge on proton and carbon NMR – Theory and spectra interpretation	NMR Spectroscopy	TB-1: 15; TB-2: 19; R2-4 & 5
24-28	Theory, various instrumental aspects and spectra analysis.	Mass Spectroscopy	TB-1: 16; TB-2: 20; R2-2
29-32	Theory and applications of DSC and TGA	Thermography	TB-1: 25; TB-2: 25
33-40	Design and development of analytical methods for the separation, purification and quantitative analysis of drugs through various chromatographic techniques	Chromatographic Techniques <ul style="list-style-type: none"> <li>• Introduction</li> <li>• Gas Chromatography</li> <li>• HPLC</li> <li>• Hyphenated LC techniques</li> <li>• High Voltage/ Gel Electrophoresis</li> </ul>	TB-1: 17; TB-2: 21 TB-1: 18; TB-2: 21 TB-1: 19,20; TB-2: 21 TB-2: 21,22; R-5 and class notes
41-42	Analysis of heavy metals and inorganic drugs	Flame photometry and A.A.S	TB-1: 9,10; TB-2: 16

**b. Plan for Laboratory:**

Laboratory sessions will be conducted so that students get hands on experience on all the sophisticated analytical instruments. Certain instruments will only be demonstrated to the students. The comprehensive list of experiments is given below.

S.No.	Name of Experiments
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<b>Experiments Involving Hands On Experience</b>	
1	<b>UV – Visible</b> Determination of $\lambda_{\text{max}}$ , verification of Beer-Lambert's Law Determination of binary mixtures Difference, derivative spectroscopy Unknowns – interpretation and analysis
2	<b>IR</b> Handling solid samples using various techniques Unknowns – interpretation, functional group analysis
3	<b>Polarimetry</b> Determination of muta-rotation in glucose, unknown / Estimation of specific rotations for a few selected drugs
4	<b>Spectrofluorimetry</b> Estimation of selected drugs compounds, unknown Quenching effect and its significance Evaluation of drug in formulations
5	<b>Paper Electrophoresis / Gel Electrophoresis</b> Instrumentation, applications – separation of proteins / cell components Identification/separation/purification of samples
6	<b>HPLC</b> Instrumentation, method development approaches. Separation and estimation of selected drug samples Unknown identification, applications
7	<b>Thermography</b> Instrumentation, calibration of DSC, TGA Unknown-identification, applications
8	<b>AAS</b> Analysis of heavy metals
<b>Demonstration Based Experiments</b>	
9	<b>Flame photometry and XRF</b> Calibration, estimation of elements, applications
10	<b>GC</b> Instrumentation, method development approaches. Separation and estimation of selected drug samples Unknown identification, applications
11	<b>LC-MS</b> Identification of unknown compounds in the mixture using MS
12	<b>NMR and Mass</b> Interpretation of spectra and characterization of organic compounds based upon IR, NMR and Mass

**Note:**

- Modifications/adjustments would be made in the theory/experimental pattern / part, if necessary as and when situation arises.
- Students should maintain and bring updated record note-books for every practical class.



- iii. Make-ups for practical are not always possible. However, depending upon the genuineness of the situations, students may be permitted to perform back-log experiments, if any instructor is free, outside regular class hours.
- iv. It is imperative that all students come prepared for the experiment in the next turn completing all pending work concerned with the previous experiment. Adequate preparation for the practical in terms of principles and operation of the instrument as per instructions, familiarization of protocols involved, outside class hours, is mandatory.

#### Evaluation Scheme:

Component	Duration	Weightage (%)	Date & Time	Nature of Component
Mid Sem Test	90 min	20	13/3 1.30 -3.00 PM	CB
Quiz (Pre and Post Midterm)	30 min	5 + 5		CB
Laboratory**	-	20		
Viva-Voce (Final)	-	10		
Compre. Exam.	3 h	40	07/05 FN	CB: 20 % OB: 20 %

#### Note:

- \*\* Laboratory component may include assignments, which will be practical or theoretical type that would include interpretation of IR, NMR, Mass spectral, Elemental data - characterization of compounds, etc., besides identification and estimation of known and unknown drugs in given samples based on experiments performed , etc.

**Chamber Consultation Hour:** To be announced in class

**Notices:** Notices concerning the course will be displayed on the Department of Pharmacy N.B. 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.

**Make-up Policy:** Make-Ups are not given as a routine. It is solely dependent upon the GENUINENESS OF THE CIRCUMSTANCES under which a student fails to appear in a scheduled evaluation component. In such circumstances, prior permission should be obtained from the Instructor-in-Charge. IN NO CASE THE MAKE-UP APPLICATION BE SLIPPED INSIDE THE CHAMBER OF THE INSTRUCTOR-IN-CHARGE. However, the decision of the Instructor-in-Charge in the above matter will be final.

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**INSTRUCTOR-IN-CHARGE**  
**Instructor -in-Charge**  
**PHA F313**

