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**Second Semester 2022-2023**

# Course Handout Part II

Date: 16-01-2023

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* : Sai Sanjay, Shreya Chauhan, Suraj Gupta, Shridula S., Velmurugan

**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, 7th 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. Yuri Kazakevich and Rosario Lobrutto. HPLC for pharmaceutical Scientists. John Wiley and Sons, Inc., N.Y., 2007

R3. A.H. Beckett and J.B. Stenlake -"Practical Pharmaceutical Analysis", Fourth Edition, CBS Pub., New Delhi., Vol. I & II, 1988.

R4. Maureen Melvin,-"Electrophoresis", John Wiley and Sons, Inc., N.Y., 1987.

R5. B.G.Nagavi,-"Laboratory Handbook of Instrumental Drug Analysis", Vallabh Prakashan, New Delhi, 1996.

R6. Mahesh, R & Others Instru. Methods of Analysis Lab Manual Notes EDD, 2007.

**Course Plan:**

1. **Lecture and Tutorial Plan**

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| --- | --- | --- | --- |
| Lect (L) and Tut. (T)  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 |
| 2-4 (L)  5-6 (T) | 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 |
| 7-9 (L)  10-11 (T) | 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 X-ray spectroscopy | X-ray spectroscopy | TB-1: 13 |
| 14-20 (L) 21-23 (T) | Knowledge on proton and carbon NMR – Theory and spectra interpretation | NMR Spectroscopy | TB-1: 15; TB-2: 19; R2-4 & 5 |
| 24-26 (L) 27-28 (T) | Theory, various instrumental aspects and spectra analysis. | Mass Spectroscopy | TB-1: 16; TB-2: 20; R2-2 |
| 29-31 (L)  32 (T) | Theory and applications of DSC and TGA | Thermography | TB-1: 25; TB-2: 25 |
| 33-37 (L) 38-40 (T) | Design and development of analytical methods for the separation, purification and quantitative analysis of drugs through various chromatographic techniques | Chromatographic Techniques   * Introduction * Gas Chromatography * HPLC * Hyphenated LC techniques, HPTLC * High Voltage/ Gel Electrophoresis | 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 |

1. **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.

|  |  |
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| **S.No.** | **Name of Experiments** |
| **Experiments Involving Hands On Experience** | |
| 1 | **UV – Visible**  Determination of λmax, verification of Beer-Lambert’s Law  Determination of binary mixtures  Difference, derivative spectroscopy  Unknowns – interpretation and analysis |
| 2 | **IR**  Handling solid samples using various techniques  Unknowns – interpretation, functional group analysis |
| 3 | **GC**  Instrumentation, method development approaches.  Separation and estimation of selected drug samples  Unknown identification, applications |
| 4 | **Spectrofluorimetry**  Estimation of selected drugs compounds, unknown  Quenching effect and its significance  Evaluation of drug in formulations |
| 5 | **Paper Electrophoresis / Gel Electrophoresis**  Instrumentation, applications – separation of proteins / cell components  Identification/separation/purification of samples |
| 6 | **HPLC**  Instrumentation, method development approaches.  Separation and estimation of selected drug samples  Unknown identification, applications |
| 7 | **Thermography**  Instrumentation, calibration of DSC, TGA  Unknown-identification, applications |
| 8 | **AAS**  Analysis of heavy metals |
| **Demonstration Based Experiments** | |
| 09 | **LC-MS**  Identification of unknown compounds in the mixture using MS |
| 10 | **NMR and Mass**  Interpretation of spectra and characterization of organic compounds based upon IR, NMR and Mass |
| 11 | **XRD** Analysis |

**Note**:

1. Modifications/adjustments would be made in the theory/experimental pattern / part, if necessary as and when situation arises.
2. Students should maintain and bring updated record note-books for every practical class.
3. 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.
4. 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 | 25 | 14/03 9.30 - 11.00AM | CB |
| Laboratory\*\* | - | 20 |  | OB |
| Comprehensive Lab Viva-Voce / Quiz | - | 10+5 |  | CB |
| Compre. Exam. | 180 min | 40 | 10/05 FN | CB+OB (50:50) |

**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 demonstrated through online.

**Chamber Consultation Hour:** After the practical class

**Notices:** Notices concerning the course will be through CMS/Google Classroom

**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.

**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.

**INSTRUCTOR-IN-CHARGE**

**Instructor -in-Charge**

**PHA F313**