Study Scheme & Syllabus of Post Graduate Diploma in Pharmaceutical Analysis

Batch 2018 onwards

By

Board of Study Pharmacy

Department of Academics

IK Gujral Punjab Technical University

First Semester

Course Code	Course Type	Course Name	Load			Marks			Credits
			L	T	P	Internal	External	Total	
PGDPA101-18	Core Theory-I	Pharmaceutical Analysis-I	4	-	-	40	60	100	4
PGDPA102-18	Core Theory-II	Pharmaceutical Analysis – II	4	-	-	40	60	100	4
PGDPA103-18	Core Theory-III	Pharmaceutical Analytical Validation	4	-	-	40	60	100	4
PGDPA104-18	Core Theory-IV	Instrumental Analytical Techniques	4	-		40	60	100	4
PGDPA105-18	Core Practical-I	Pharmaceutical Analysis-I Lab	-	-	4	60	40	100	2
PGDPA106-18	Core Practical-II	Instrumental Analytical Techniques Lab		-	4	60	40	100	2
PGDPA107-18	Skill Enhancement	Seminar/Journal Club	4	-	-	100	-	100	4
PGDPA108-18	Skill Enhancement	Project-I	-	-	4	-	100	100	2
		Total	**20**		**12**	**380**	**420**	**800**	**26**

Second Semester

Course Code	Course Type	Course Name	Load			Marks			Credits
			L	T	P	Internal	External	Total	
PGDPA201-18	Core Theory-V	Advanced Pharmaceutical Analysis-I	4	-	-	40	60	100	4
PGDPA202-18	Core Theory-VI	Advanced Pharmaceutical Analysis-II	4	-	-	40	60	100	4
PGDIPR203-18	Core Theory-VII	Intellectual Property Rights& Documentation	4	-	-	40	60	100	4
PGDPA204-18	Core Practical-III	Advanced Pharmaceutical Analysis Lab	-	-	4	60	40	100	2
PGDPA205-18	Skill Enhancement	Seminar/Journal Club	4	-	4	100	-	100	2
PGDPA206-18	Skill Enhancement	Industrial Training*	-	-	-	50	50	100	4
PGDPA207-18	Skill Enhancement	Project-II	-	-	12	-	200	200	6
		Total	**16**		**16**	**330**	**470**	**800**	**28**

*Industrial Training of two weeks duration will be undertaken after 1st semester and beforecommencement of 2nd semester

1st SEMESTER

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 101-18	Pharmaceutical Analysis-I	4	-	-	40	60	1.5	3	4

Scope: This course deals with the fundamentals of analytical chemistry and titrimetric principles of analysis of drugs

Course Outcome: Upon completion of the course student shall be able to
1. Understand the principles of volumetric analysis
2. Carry out various volumetric titrations
3. Develop analytical skills

Module 01 **06 Hrs**

Quantitative Analysis and Data Handling: Introduction and Significance of quantitative analysis in quality control, different techniques of analysis, preliminaries and definitions, significant figures. Rules for retaining significant figures, Types of errors (Determinate and Indeterminate). Minimization of errors, Propagation of errors in addition and subtraction, multiplication and division, exponents, logarithms, precision and accuracy, selection of sample.

Module 02 **12 Hrs**

Acid Base Titrations: Acid base concept, role of the solvent, Relative strengths of acids and bases; Law of mass action; common ion effect, ionic product of water, pH, Hydrolysis of salts, Handerson – Hasselbach equation; Buffer and buffer capacity: Acid base indicators, Theory of indicators, Choice of indicators; Neutralization curves (Strong acid and strong base, strong acid weak base, weak acid strong base and weak acid weak base).

Polyprotic system, dissociation calculations for polyprotic acids, fractions and equilibrium concentrations of dissociating species at a given pH, salts of polyprotic acids, (Amphoteric salts and unprotonated salts), Buffer calculations for polyprotic acids, titrations of polyprotic acid, amino acid system and its titrations. Application in assay of H_3BO_3, HCl, NaOH and Na_2CO_3.

Module 03 **12 Hrs**

Oxidation-Reduction Titrations: Concepts of oxidation and reduction, redox reactions, equivalent weights of oxidizing and reducing agents, electrochemical cells, reduction potential, standard reduction potential, Nernst equation, cell representations, measurement of electrode potential and its application in determining the equilibrium constant of a reaction.

Oxidation reduction curves, redox indicators, potassium pemanganate titrations, iodimetry and iodometry, ceric sulphate titrations, potassium iodate titrations, sodium 2, 6-dichlorophenol – indophenols titrations, pharmaceutical applications.

Module 04 **08 Hrs**

Precipitation Titrations: Precipitation reactions, solubility product, effects of common ion, acids, temperature and solvent upon the solubility of a precipitate, conditional solubility product, fractional precipitation.
Argentometric titrations, ammonium or potassium thiocyanate titrations, mercuric nitrate titrations, indicators, Gay-Lussac method, Mohr's method, Volhard's method, Fajan's method, Pharmaceutical applications.

Module 05 **07 Hrs**

Gravimetric Analysis: Precipitation techniques, the colloidal state, gravimetric factor, supersaturation, co-precipitation and its types, Post precipitation, digestion, washing of the precipitate, filtration, filter papers and crucibles, ignition, thermogravimetric curves of copper sulphate, specific examples like barium as barium sulphate, aluminium as aluminium oxide, calcium as calcium oxalate and magnesium as magnesium pyrophosphate, organic precipitants.

Recommended Books (Latest Edition)

1. Becket & Stenlake. Practical Pharmaceutical Chemistry. Vol. 1& 2.. CBS Publishers, New Delhi.
2. Jeffery, Bassett & Mendham. Vogel’s text book of Quantitative chemical analysis. Addison Wesley Longman Ltd England.
3. Danzer K, Analytical Chemistry, Springer.
4. R.M. Verma. Analytical Chemistry. CBS Publishers, New Delhi.
5. Alexeyev. Qualitative Analysis. CBS Publishers, New Delhi.
6. L. M. Atherden, Bentley and Driver's Textbook of Pharmaceutical Chemistry, Oxford University Press, Delhi.

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 102-18	Pharmaceutical Analysis –II	4	-	-	40	60	1.5	3	4

Scope: This course deals with the analysis of drugs by different methods like titrimetric analysis, extraction, radioactivity, chromatography and electrochemical methods.

Course Outcome:

Upon completion of the course student shall be able to do

1. The analysis and isolation of various drugs from mixture by various methods.
2. Theoretical and practical skills of the instruments
3. Understand the basic principle behind various analytical techniques

Module 01 **06 Hrs**

Non- aqueous Titrations: Theoretical consideration, scope and limitations, acid base equilibria in non-aqueous media, titration of weak bases, titration of weak acids, indicators, pharmaceutical products should be selected for illustration.

Module 02 **06 Hrs**

Complexometric Titrations: Concept of complexation and chelation, Werner's Coordination number and electronic structure of complexions, stability constants, titration curves, masking and demasking agents, types of Complexometric titrations, metal ion indicators, factors influencing the stability of complexes, applications.

Module 03 **06 Hrs**

Miscellaneous Methods of Analysis: Diazotisation titration, Kjeldahl nitrogen determination, Karl- Fischer titration, Oxygen flask combustion. Extractions Procedures: Separation of drugs from excipients, The Craig method of multiple extraction, continuous counter - current extraction, effect of temperature, pH, inert solute, association, ion-pair formation, the emulsion problems in extractions.

Module 04 **08 Hrs**

Nuclear Chemistry and Radioactivity as an Analytical Tool: Nuclear composition, forces and stability, isotopes, radioactive emission, measurement of radioactivity, modes of decay, half-life period, artificial radioactivity, applications in pharmacy.Radiopharmaceutical and contrast media: Radio-pharmaceuticals, radiopharmaceutical preparations and radiopaque contrast media, counting statistical errors and corrections, safety.

Module 05 **09Hrs**

Chromatography: Gas chromatography: Introduction; Principles of gas chromatography, basic GLC apparatus, carrier gases; sample introduction, column, column efficiency, solid support, liquid phases, branches of gas chromatography; Detectors, temperature effect; Applications of GLC in Pharmaceutical analysis.

Ion-Exchange Chromatography: Theory of ion exchange, types of exchangers, ion exchange equilibria, ion-exchange capacity, ion-exchange separation, applications in pharmaceutical analysis, molecular sieve separation and applications.

Module 06 **10 Hrs**

Electrochemistry: The electric cell, electrode potential, half cells, types of half cells, sign convention, Nernst equation, the salt bridge, activity series, standard potential, standard hydrogen electrode, measuring the relative voltage of half cells, calculations of standard potential, reference electrodes, indicator electrodes.

a. Potentiometry: Theoretical consideration, ion-selective electrodes, measurement of potential, location of the end point, equipment, analytical applications, direct measurement of a metal concentration, differential curves, pH measurements, dead-stop titrations; pH meter, pH definition, relation of pH to potential, equipment, applications.
b. Conductometric and High Frequency Titrations: Principles and their Applications.
c. Coulometric Titrations: Its basic principles and Applications.
d. Polarography and its applications: Theory, mass transport processes, current processes, current potential relationship, polarization, choice of electrodes, effect of oxygen, instrumentation, calculation of concentration, laboratory design and safety.
e. Amperometric Titrations and its Applications

Recommended Books (Latest Edition)

1. A.H. Beckett and J.B. Stenlake, Practical Pharmaceutical Chemistry, Vol. I & II, The Athlone Press of the University of London.
2. J. Bassett, R.C. Denney, G.H. Jeffery & J. Medhan, Vogel's Textbook of Quantitative Inorganic Analysis Including Elementary Instrumental Analysis. The English Language Book Society and Longman.
3. H. H. Willard, L.L. Merritt; Jr., and J.A. Dean, Instrumental Methods of Analysis, Van Nostrand Reinhold, New York.
4. L. G. Chatten, Pharmaceutical Chemistry, Vols. I and II, Marcel Dekker, New York.
5. Braun, Introduction to Instrumental Analysis, I edition, PharmaMed Press Danzer, K., Analytical Chemistry Theoretical and Metrological Fundamentals, Springer.

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 103-18	Pharmaceutical Analytical Validation	4	-	-	40	60	1.5	3	4

Scope: The main purpose of the subject is to understand about validation and how it can be applied to industry and thus to improve the quality of the products. The subject covers the complete information about validation, types, methodology and application.

Course Outcomes: Upon completion of the course, student shall be able to

1. Explain the aspect of validation.
2. Explain the aspect of validation.
3. Apply the knowledge of validation to instruments and equipment's.
4. Validate the manufacturing facilities

Module 01 **10 Hrs**

Introduction, Definition of Qualification and Validation, Advantage of Validation, Streamlining of Qualification & Validation process, Validation Master Plan Qualification, User Requirement Specification, Design Qualification, Factory Acceptance Test (FAT)/ SiteAcceptance Test (SAT), Installation Qualification, Operational Qualification, PerformanceQualification, Re-Qualification (Maintaining status- Calibration Preventive Maintenance,Change management), Qualification of Manufacturing equipments, Qualification of Analytical Instruments and Laboratory equipments.

Module 02 **07 Hrs**

Qualification of Analytical Instruments: Electronic balance, pH meter, UV-Visible spectrophotometer, FTIR, GC, HPLC, HPTLC, Qualification of Glassware: Volumetric flask, pipette, Measuring cylinder, beakers and burette.

Module 03 **10 Hrs**

Validation of Utility Systems: Pharmaceutical Water System & pure steam, HVAC system, Compressed air and nitrogen Cleaning Validation. Cleaning validation - cleaning method development, validation and validation of analyticalmethod used in cleaning, Cleaning of Equipment, cleaning of facilities, cleaning in place (CIP).

Module 04 **10 Hrs**

Analytical Method Validation: General principles, Validation of analytical method as per ICH guidelines and USP. Computerized System Validation: Electronic records and digital significance-21 CFR part 11 and GAMP 5

Module 05 **08 Hrs**

Extraction of Drugs and Metabolites from Biological Matrices: General need, principle and procedure involved in the Bioanalytical methods such as Protein precipitation, Liquid - Liquid extraction and Solid phase extraction and other novel sample preparation approach. Bioanalytical Method Validation: USFDA and EMEA guidelines.

Recommended Books (Latest Edition)

1. B. T. Loftus & R. A. Nash, "Pharmaceutical Process Validation, Drugs and Pharm Sci. Series, Vol. 129, Marcel Dekker Inc., N.Y.
2. The Theory & Practice of Industrial Pharmacy, Leon Lachman, Herbert A. Lieberman, Joseph, L. Karig, Varghese Publishing House, Bombay.
3. Validation Master Plan by Terveeks or Deeks, Davis Harwood International publishing.
4. Validation of Aseptic Pharmaceutical Processes, by Carleton &Agalloco, Marcel Dekker.
5. Michael Levin, Pharmaceutical Process Scale-Up, Drugs and Pharm. Sci. Series, Vol. 157,
6. Marcel Dekker Inc., N.Y. Validation Standard Operating Procedures: A Step by Step Guide for Achieving Compliance in the Pharmaceutical, Medical Device, and Biotech Industries, Syed Imtiaz Haider.
7. Pharmaceutical Equipment Validation: The Ultimate Qualification Handbook, Phillip A. Cloud, Interpharm Press.
8. Validation of Pharmaceutical Processes: Sterile Products, Frederick J. Carlton (Ed.) and James Agalloco (Ed.), Marcel Dekker.
9. Analytical Method validation and Instrument Performance Verification by Churg Chan, Heiman Lam, Y.C. Lee, Yue. Zhang, Wiley Inter Science.

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 104-18	Instrumental Analytical Techniques	4	-	-	40	60	1.5	3	4

Scope: This subject deals with the application of instrumental methods in qualitative and quantitative analysis of drugs. This subject is designed to impart advanced knowledge on the principles and instrumentation of spectroscopic and chromatographic hyphenated techniques. This also emphasizes on theoretical and practical knowledge on modern analytical instruments that are used for drug testing.

Objectives: Upon completion of the course, the student shall be able to know

1. Analysis of drugs using various analytical instruments
2. Understand the chromatographic separation and analysis of drugs
3. Understand the advanced instruments used and its applications in drug analysis

Module 01 **13 Hrs**

UV-Visible Spectroscopy: Introduction, Theory, Laws, and Instrumentation, Choice of solvents and solvent effect, Applications of UV- Visible spectroscopy, Difference/ Derivative spectroscopy

IR Spectroscopy: Theory, Modes of Molecular vibrations, Sample handling, Instrumentation of Dispersive and Fourier - Transform IR Spectrometer, Factors affecting vibrational frequencies, Applications of IR spectroscopy, Data Interpretation.

Spectroflourimetry: Theory of Fluorescence, Factors affecting fluorescence (characteristics of drugs that can be analysed by flourimetry), Quenchers, Instrumentation, Applications of fluorescence spectrophotometer

Flame Emission Spectroscopy and Atomic Absorption Spectroscopy: Principle, Instrumentation, Interferences and Applications

Module 02 **10 Hrs**

Chromatography: Principle, apparatus, instrumentation, chromatographic parameters, factors affecting resolution and applications of the following:

- Thin Layer chromatography
- High Performance Thin Layer Chromatography
- Ion exchange chromatography
- Gas chromatography
- High Performance Liquid chromatography
- Affinity chromatography
- Gel Chromatography

Module 03 **12 Hrs**

HPLC: Principle, instrumentation, pharmaceutical applications, Peak shapes, capacity factor, selectivity, plate number, plate height, resolution, band broadening, Pumps, injector, detectors, columns, column problems.

Gradient HPLC, HPLC solvents, trouble shooting, sample preparation, method development, New developments in HPLC-role and principles of ultra, nano liquid chromatography inpharmaceutical analysis.

Preparative HPLC, practical aspects of preparative HPLC

Module 04 **10 Hrs**

Electrophoresis: Principle, Instrumentation, working conditions, factors affecting separation and applications of the following:

- Paper electrophoresis
- Gel electrophoresis
- Capillary electrophoresis
- Zone electrophoresis
- Moving boundary electrophoresis
- Isoelectric focusing

X ray Crystallography: Production of X rays, Different X ray diffraction methods, Bragg's law, Rotating crystal technique, X ray powder technique. Types of crystals and applications of X-ray diffraction

Recommended Books (Latest editions)

1. Spectrometric Identification of Organic Compounds - Robert M Silverstein, John Wiley & Sons.
2. Principles of Instrumental Analysis - Doglas A Skoog, F. James Holler, Timothy A. Nieman, Eastern press, Bangalore.
3. Instrumental methods of analysis – Willards, CBS publishers.
4. Practical Pharmaceutical Chemistry – Beckett and Stenlake, Vol II, CBS Publishers, New Delhi.
5. Organic Spectroscopy - William Kemp, ELBS.
6. Quantitative Analysis of Drugs in Pharmaceutical formulation - P D Sethi, CBS Publishers, New Delhi.
7. Pharmaceutical Analysis- Modern methods – Part B - J W Munson, Volume 11, Marcel Dekker Series.
8. Spectroscopy of Organic Compounds, P.S. Kalsi, Wiley Estern Ltd., Delhi.
9. Textbook of Pharmaceutical Analysis, K. A. Connors, John Wiley & Sons.

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 105-18	Pharmaceutical Analysis-I Lab	-	-	4	60	40	6	6	2

1. To perform calibration of volumetric apparatus and weights including fractional weight using digital weighing balance of sensitivity 1 mg.

2. Preparation and Standardization of
   - Sodium hydroxide
   - Sulphuric acid
   - Sodium thiosulfate
   - Potassium permanganate
   - Ceric ammonium sulphate

3. Assay of the following compounds along with standardization of titrant

   - Ammonium chloride by acid base titration
   - Ferrous sulphate by Cerimetry
   - Copper sulphate by Iodometry
   - Calcium gluconate by complexometry
   - Hydrogen peroxide by Permanganometry
   - Sodium benzoate by non-aqueous titration
   - Sodium Chloride by precipitation titration

4. Determination of Normality by Electro-Analytical Methods

   - Conductometric titration of strong acid against strong base
   - Conductometric titration of strong acid and weak acid against strong base
   - Potentiometric titration of strong acid against strong base

5. Preparations and standardization of EDTA solution, some exercises related to pharmacopoeial assays by complexometric titrations.

6. Preparation and standardization of perchloric acid and estimations of some pharmacopoeial products.

Recommended Books (Latest Edition)

1. Jeffery, Bassett & Mendham. Vogel text book of quantitative chemical analysis. Addison Wesley Longman ltd England.
2. R.M.Verma. Analytical chemistry. CBS Publishers, New Delhi. Becket& Stenlake. Practical pharmaceutical chemistry. Vol.1 & 2. CBS publishers, New Delhi.

3. Alexeyev. Quantative analysis. CBS publishers, New Delhi.
4. L.M.Atherden, Bentley and Driver's textbook of pharmaceutical chemistry, Oxford University Press, Delhi.
5. A.H. Beckett and J.B. Stenlake's, Practical Pharmaceutical Chemistry Vol I and II, Stahlone Press of University of London.
6. Indian Pharmacopoeia.

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 106-18	Instrumental Analytical Techniques Lab	-	-	4	60	40	6	6	2

1. Analysis of Pharmacopoeial compounds and their formulations by UV Vis spectrophotometer
2. Simultaneous estimation of multi component containing formulations by UV Spectrophotometry
3. Comparison of absorption spectra by UV and Wood ward – Fiesure rule
4. Interpretation of organic compounds by FT-IR
5. Experiments based on HPLC
6. Experiments based on Gas Chromatography
7. Estimation of riboflavin/quinine sulphate by fluorimetry
8. Estimation of sodium/potassium by flame photometry
9. Calibration of UV-Visible spectrophotometer
10. Calibration of FTIR spectrophotometer
11. Calibration of GC instrument
12. Calibration of HPLC instrument

Recommended Books (Latest Editions)

1. Instrumental Methods of Chemical Analysis by B.K Sharma.
2. Organic spectroscopy by Y.R Sharma.
3. Vogel's Text book of Quantitative Chemical Analysis by A.I. Vogel.
4. Practical Pharmaceutical Chemistry by A.H. Beckett and J.B. Stenlake.
5. Quantitative Analysis of Drugs in Pharmaceutical Formulations by P. D. Sethi.
6. Spectrophotometric identification of Organic Compounds by Silverstein.

Course Code	Course Title	Teaching Load			Marks		Exam (hrs)		Credits
		L	T	P	Int.	Ext.	Int.	Ext.	
PGDPA 108-18	Project-I	-	-	4	100	-	1	-	2

The candidate shall prepare a report on a topic of recent development assigned by the mentor. The candidate shall also give a presentation to be evaluated by both internal and external examiners.

Evaluation Scheme

Report : 25 marks

Presentation : 25 marks

Viva-Voce : 50 marks