M.PHARMACY  
PHARMACEUTICAL ANALYSIS AND QUALITY ASSURANCE  

I SEMESTER  
Paper 101 - Modern Analytical Techniques  
Paper 102 - Research Methodologies  
Paper 103 - Advanced Pharmaceutical Analysis - I  
Paper 104 - Chromatographic and Other Special techniques  
Paper 105 - Advanced Pharmaceutical Analysis-I - LAB  
Paper 106 - Chromatographic and Other Special techniques - LAB  
Paper 107 - Seminar  

II SEMESTER  
Paper 201 - Advanced Pharmaceutical Analysis - II  
Paper 202 - Phytopharmaceutical and Biological Analysis  
Paper 203 - Quality Assurance of Pharmaceuticals – I  
Paper 204 - Drug Regulatory Affairs  
Paper 205 - Advanced Pharmaceutical Analysis - II - LAB  
Paper 206 - Phytopharmaceutical and Biological Analysis - LAB  
Paper 207 - Seminar  

III SEMESTER  
Paper 301 - Project Seminar-I (On the proposed project work with aims and objectives) - 50 Marks  
Paper 302 - Project work - I  

IV SEMESTER  
Paper 401 - Project Seminar-II (On the experimentation and results of the project work) – 50 Marks  
Paper 402 - Project work - II
## SCHEME OF INSTRUCTIONS AND EVALUATION

### PHARMACEUTICAL ANALYSIS AND QUALITY ASSURANCE

#### FIRST SEMESTER

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## SECOND SEMESTER

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| Credits | 18 |

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## THIRD AND FOURTH SEMESTERS

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M.PHARM SYLLABUS FOR PHARMACEUTICAL ANALYSIS AND QUALITY ASSURANCE

I SEMESTER

PAPER 101: MODERN ANALYTICAL TECHNIQUES
(Paper Common for all Specializations)

Principles, instrumentation and applications of the following Instruments and Chromatography techniques

**Unit- I**
- i. UV- Visible spectrophotometry
- ii. Infrared spectroscopy
- iii. Spectrofluorimetry

**Unit- II**
- i. NMR spectroscopy
- ii. Electron Spin Resonance spectroscopy
- iii. Atomic Emission spectroscopy

**Unit- III**
- i. HPLC
- ii. HPTLC
- iii. Exclusion chromatography
- iv. Super critical fluid chromatography

**Unit- IV**
- i. Mass Spectroscopy including LCMS & GCMS
- ii. GLC

**Unit- V**
- i. Plasma Emission spectroscopy
- ii. X-Ray diffractometry
- iii. Optical Rotatory Diffusion
- iv. Vapor phase chromatography
- v. Affinity chromatography
- vi. Ion-exchange chromatography

**TEXT BOOKS**
1. Practical Pharmaceutical Chemistry Vol. 1 &II by Beckett & Stenlake.
2. Instrumental Methods of Analysis by Scog and West.
3. Instrumental Methods of Analysis by B.K.Sharma
5. Instrumental methods of Analysis by Willard & Merrit.

**REFERENCE BOOKS**
1. I.P.
2. B.P.
UNIT I
Statistical Methods:
Chance Variation – Probability Distribution - Normal Distribution – Sampling Distribution
Error and its significance-Measures of Error- Control of Error in Experimental Investigations –
Problem Solving.

UNIT II
Correlation and Regression., Multiple Regression - Problem Solving

UNIT III
Tests of Significance: Principles, t-test, z-test, F-ratio test, Chi-square test, Non-parametric
tests- their applications in pharmacy research with examples – Problem Solving

UNIT IV
Design of Experiments
Criteria of a good design with examples.
Principles- Randomization, replication and local control.
Study of CRD, RBD, LSD and factorial designs- their applications in Pharmacy research with
examples – Problem Solving

UNIT V
Analysis of Variance (ANOVA) – one way, two way and three way – principles and
applications in pharmacy research- Problem Solving
Optimisation Techniques: Optimisation Techniques based on Factorial Experiments - Problem
Solving.

Recommended Books:
1. Fundamentals of Biostatistics by Khan & Khanum, Ukaaz Publications, Hyderabad
2. Theory & Practice of Industrial Pharmacy by Leon Lachman and Others
4. Principles of Biostatistics by Marcello Pagnano, Published by Brooks/Cole, (Saurabh
Printers Pvt. Ltd)
UNIT-I
1. Good Laboratory practices (GLP), Laboratory maintenance, standard operating procedures (SOPS), Validation of analytical instruments and methods. – Quality Control Laboratory Regulatory requirements

UNIT-II
1. Theory, Instrumentation and application with regard to drug analysis, decomposition product identification and estimation and metabolite analysis based on the following:
   a) Ultraviolet visible spectrophotometry
   b) Infrared Spectrophotometry
   c) Fluoresmetry, Nephelometry and Turbidimetry

UNIT-III
1. Polarography.
2. Flame emission spectroscopy and atomic absorption spectroscopy. Principle, Instrumentation and applications in Pharmacy.

UNIT-IV
1. Thermal methods of analysis: Theory of Thermo gravimetric analysis (TGA), Differential Thermal analysis (DTA), Differential Scanning Calorimetry (DSC) and Thermo Mechanical Analysis (TMA).
2. An advanced study of non-aqueous titrations involving the following:
   a) Primary, Secondary and Tertiary amines
   b) Halogenated salts and bases
   c) Acidic substances
   d) Assays of official drugs in IP 1996 by non-aqueous titrimetry
   e) Aquametry: Determination of water by titration with Karl Fischer Reagent (KFR).

UNIT-V
1. Principles and pharmaceutical applications of redox titrations involving:
   a) Potassium iodate / bromate titrations
   h) Ceric ammonium sulphate titrations
c) Tanus Chloride titration
d) Examples of assays of official drugs in IP 1996.
2. Principles and Pharmaceutical applications of complexometric titrations involving:
   a) Direct titration of Polymetallic system with Sodium EDTA
   b) Back titration with sodium EDTA
   c) titration involving the displacement of one complex by another
d) PM indicators
e) Examples of assays official drugs in IP 1996.
TEXT BOOKS
1. Practical Pharmaceutical Chemistry Vol. 1 &II by Beckett & Stenlake.
2. Instrumental Methods of Analysis by Scog and West.
3. Instrumental Methods of Analysis by B.K.Sharma
5. Instrumental methods of Analysis by Willard & Merrit.

REFERENCE BOOKS
1. I.P.
2. B.P.
3. U.S.P.
5. Spectroscopy b Silversterin
PAPER 104: CHROMATOGRAPHIC AND OTHER SPECIAL TECHNIQUES

UNIT-I
An advanced study of the following and their applications.
1. Basic principle and separation by Column chromatography, thin layer chromatography, paper chromatography and ion exchange chromatography.

UNIT-II
1. Gas Chromatography: Introduction, theory, column operation, instrumentation and detection, GCMS.

UNIT-III
1. High Pressure Liquid Chromatography: Principle, Instrumentation procedure, solvents used, elution techniques, LCMS and applications.

UNIT-IV
1. HPTLC and Supercritical Fluid Chromatography (SFC): Principle, instrumentation procedure, elution technique and pharmaceutical applications.
2. H.P.C.P.C

UNIT-V
1. Electrophoreses (gel and capillary)
2. Radio immuno assay and related immuno assays — RIA, ELISA

TEXT BOOKS
1. Instrumental Methods of Analysis by Scog and West.
2. Instrumental Methods of Analysis by B.K.Sharma
3. Instrumental methods of Analysis by Willard & Merrit.

REFERENCE BOOKS
1. USP
2. Remington’s Pharmaceutical Sciences.
3. Spectroscopy by Silversterin
PAPER 105: ADVANCED PHARMACEUTICAL ANALYSIS –I

1. Use of spectrophotometer for analysis of Pharmacopoeial compounds and their formulations.
2. Use of fluorimeter for analysis of Pharmacopoeial compounds.
3. Use of Flame photometer for analysis of Na, K & Ca etc in Biological fluids and formulations.
4. Use of Nephelo-Turbidimetric analysis of dispersions and limit tests.
5. Assays involving following procedures: Non – Aqueous, Diazotisation, Complexation and Redox titrations.

TEXT BOOKS
1. Practical Pharmaceutical Chemistry Vol. 1 &II by Beckett & Stenlake.
2. Instrumental Methods of Analysis by Scog and West.
3. Instrumental Methods of Analysis by B.K.Sharma
5. Instrumental methods of Analysis by Willard & Merrit.

REFERENCE BOOKS
1. I.P.
2. B.P.
3. U.S.P.
5. Spectroscopy b Silversterin

TEXT BOOKS
1. Instrumental Methods of Analysis by Scog and West.
2. Instrumental Methods of Analysis by B.K.Sharma
3. Instrumental methods of Analysis by Willard & Merrit.

REFERENCE BOOKS
1. USP
2. Remington’s Pharmaceutical Sciences.
3. Spectroscopy by Silversterin
PAPER 106: CHROMATOGRAPHIC AND OTHER SPECIAL TECHNIQUES LAB

1. Experiments on Electrophoresis.
2. Experiments of Chromatography:
   a) Ascending technique
   b) Descending technique
   c) Circular technique
3. Experiments using HPLC & GC.

TEXT BOOKS
1. Instrumental Methods of Analysis by Scog and West.
2. Instrumental Methods of Analysis by B.K. Sharma
3. Instrumental methods of Analysis by Willard & Merrit.

REFERENCE BOOKS
1. USP
2. Remington’s Pharmaceutical Sciences.
3. Spectroscopy by Silversterin
II SEMESTER

PAPER 201 - ADVANCED PHARMACEUTICAL ANALYSIS-II

UNIT-I
1. A detailed study of the principles, instrumentation and applications of the following instrumental analysis:
   i. Nuclear magnetic resonance spectrometry - \(^1\)H NMR ,2D NMR, COSY, \(^{13}\)CNMR, DEPT Experiments
   ii. Mass spectroscopy.

UNIT-II
1. A detailed study of the principles, instrumentation and applications of the following instrumental analysis:
   i. X-ray fluorescence spectrometry
   ii. Raman Spectroscopy
   iii. Inductively coupled plasma - atomic emission spectroscopy
   iv. Electron spin resonance spectroscopy (ESR)

UNIT-III
1. A detailed study of the various principles and procedures involved in the qualitative and quantitative analysis of pharmaceutical preparations and dosage forms containing the following groups of drugs included in IP (Biological and microbiological methods excluded)
   a) Analgesics and antipyretics
   b) I3arbiturates
   c) Sulphonamidcs
   d) Antibiotics
   e) Steroidal hormones
   f) Vitamins
   g) Alkaloids

UNIT-IV
1. A detailed study of the principles and procedures involved in the qualitative and quantitative analysis of pharmaceutical preparations and dosage forms using the following reagents and reactions.
   i) Oxidative coupling reactions using MBTH (3 - methyl -2 benzothiazolinone hydrazone hydrochloride)
   ii) Diazotisation followed by coupling
   iii) Oxidation followed by complexation.

2. A detailed study of the principles and procedures involved in the qualitative and quantitative analysis of pharmaceutical preparations and dosage form using the following reagents and reactions
   i) Oxidation followed by charge transfer reaction.
   ii) Condensation reactions using the reagents Para Dimethyl Amino Benzaldehyde (PDAB), Para Dimethyl Amino Cinnamaldehyde (PDAC), Folin’s reagent and Gibb’s reagent
   iii) Folin-ciocalteu reagent (FC reagent)
UNIT- V

2. Testing of containers and closures (glass, metal, rubber and plastic) for pharmaceutical preparations as per the I.P.

TEXT BOOKS
1. Instrumental methods of analysis by Scog and West.
2. Chemical Analysis - Modem Instrumentation methods and techniques by Wiley.
3. Instrumental methods of analysis by Willard Dean & Merrit.
5. Pharmaceutical analysis edited by Highuchi and Brochman

REFERENCE BOOKS
3. IP
4. BP
5. USP
PAPER 202 - PHYTOPHARMACEUTICAL AND BIOLOGICAL ANALYSIS

UNIT-I
1. Methods of systematic phytochemical analysis including extraction and identification of constituents using chromatographic techniques.
2. Quality control of crude drugs: proximate analysis including ash and extractive values, fiber content, U.V and fluorescence analysis of powdered drugs.

UNIT-II
1. Qualitative and quantitative microscopy and chemical microscopy and micro chemical tests.
2. Detection of common adulterants and insects infestation in whole and powdered drugs.

UNIT-III

UNIT-IV
1. Analysis of official formulations derived from crude drugs including some ayurvedic preparations.
7. Microbiological screening methods for antimicrobial activity.

UNIT-V
8. Official (IP) Bio assays and Toxicity studies as per IP 1985: Test for histamine like substances, test for pyrogens, test for undue toxicity, Acute, Sub acute and Chronic Toxicity Studies

TEXT BOOKS
1. Textbook of Pharmacognosy by Trease & Evans.
2. Textbook of Pharmacognosy by Titler, Brady & Robber.
3. Phytochemical methods by J.B.Harborne.
4. Instrumental methods of Analysis by Willard, Meritt, Dean.
4. The Quantitative analysis of Drugs by D.C.Garat
5. Microbiological assays by Barton J.Wright.

REFERENCE BOOKS
1. Pharmacopoeia of India
2. Pharmacopoeial standards for ayurvedic Formulation (Council of Research in Medicine & Homeopathy)
4. Analytical Microbiology by Kavanaugh.F
UNIT-I
1. Concept of Quality assurance, total quality management, philosophy of GMP, CGMP and GLP.
2. Organization and personnel, responsibilities, training hygiene - Premises: Location, design, plan layout, construction, maintenance and sanitations, environmental control, sterile areas, control of contamination.

UNIT-II

UNIT-III
1. Manufacture and controls on dosage forms, manufacturing documents master formula, batch formula records, standard operating procedures, quality audits of manufacturing processes and facilities - In process quality control on various dosage forms: sterile, biological products and non sterile, standard operating procedures for various operations like cleaning, filling, drying, compression, coating, disinfection, sterilization, membrane filtration etc.
Guidelines for Quality Assurance of Human Blood products and large volume parenterals.
2. Packaging and labeling controls, line clearance and other packaging materials.

UNIT-IV
1. Quality Control Laboratory: Responsibilities, good laboratory practices, routine controls, instruments, protocols, non-clinical testing, controls on animal house, data generation and storage, quality control documents, retention samples, records, audits of quality control facilities - Finished products release: quality review, quality audits, and batch release document.

UNIT-V
1. Distribution and Distribution records: Handling of returned goods, recovered materials and reprocessing.
2. Complaints and recalls, evaluation of complaints, recall procedures, related records and documents.

TEXT BOOKS
3. GMP-Mehra
4. Pharmaceutical Process validation by Berry and Nash
REFERENCE BOOKS
2. Basic tests for Pharmaceutical substances - WHO (1991)
3. How to practice GMP’s – P.P.Sharma
4. The Drugs and Cosmetic Act 1940- Vijay Malik
5. Q.A Manual by D.H.Shah
6. SOP Guidelines by D.H.Shah
7. Quality Assurance Guide by OPPI
PAPER 204: DRUG REGULATORY AFFAIRS:
(Paper Common for all Specializations)

Unit - I
Formulation development: Regulatory requirements involved in the preformulation studies, solid, liquid and semi-solid dosage forms, controlled release preparations, injections, ocular preparations as per the European community, United States and Indian regulatory authorities

Unit - II
Manufacturing: Regulatory requirements as per European community, United States and Indian regulatory authorities for manufacturing information, manufacturing formula, process, validation of manufacturing process, equipment, documentation, inspection requirement of regulatory guidelines for active ingredients, data requirement for new drug, International aspects of Excipients, approval as per guidelines of all the territories. Regulatory guidelines for packaging materials, test and evaluation of packaging materials, biological test, elastometer test, microbiological test and evaluation of closures.

Unit - III
Stability testing: Scientific and technical background to the design of stability testing regulatory requirements as per European community, United States and Indian regulatory authorities for testing of new active substances, bulk active drug substances, dosage form in their final packaging. Extension of shelf-life after authorization of drug international harmonization and current guidelines. Regulatory affairs in respect of residual solvents as per the ICH guidelines, analytical method validation, pharmacokinetic and toxicokinetic validation.
Biopharmaceutics: Different testing parameters and standards as per regulatory requirements of European community, United States and Indian regulatory authorities with respect to factors related to formulation, dosage form, manufacturing process, stability and storage.

Unit - IV
Preclinical aspects of Biopharmaceutics: Current guidelines and developments as per regulatory requirements of European community, United States and Indian regulatory authorities in respect of clinical bioavailability, study design, presentation documentation and statistical analysis
Clinical pharmacology and Pharmacodynamics: Regulatory guidelines as per European community, United States and Indian regulatory authorities on clinical study design, documentation, presentation and interpretation. Clinical trials: Definition, phase I, phase II, phase III and phase IV studies, design documentation, presentation and interpretation, statistical analysis of clinical data and factorial design.

Unit - V
Intellectual property rights and patents: Introduction, purpose, international scenario and Indian scenario, guidelines as per European community, United States and Indian regulatory authorities, documentation, presentation and application, procedure for obtaining and writing a patent and patenting rules and regulations

**References:**
1. Quality Assurance Guide by Organization of Pharmaceutical producers of India.
5. Pharmaceutical Preformulations by J.J. Wells.
6. Applied production and operations management by Evans, Anderson, Sweeney and Williams.
1. Estimation of following classification of drugs using different analytical methods.
   a) Analgesics and Antipyretics b) Barbiturates c) Sulfonamide drugs
d) Antibiotics e) Steroidal hormones f) Vitamins
g) Alkaloids

2. Estimation of different classification of drugs using the following reagents:
   a) MBTH  b) PC reagent  c) FeCl₃ and 1,10- phenanthroline
d) FeCl₃ & K₃ Fe (CN)₆  e) BM reagent  f) p-dimethylamine benzaldehyde
g) p-dimethylamino cinnamaldehyde  h) N-bromo succinimide-metol/sulphanilamide.

3. Quality control test for official formulations.
4. Testing of containers and closures (glass, metal, rubber and plastic) for official (IP) pharmaceutical preparations.
1. Spectrophotometric determination of caffeine from tea powder.
2. The estimation of curcumin from Curcuma longa by Spectrophotometric methods.
3. Determination of sugars by descending paper chromatography.
4. Determination of bitterness value of crude drugs.
5. Determination of extractive values of crude thugs.
7. Determination of $R_f$ values of different amino acids and alkaloids.
8. Anti-microbial activity of some plant extracts using different pathogenic and non-pathogenic organisms.
9. Colorimetric analysis of some plant drugs.
11. Screening for analgesic and anti-inflammatory activities.