# M. PHARM SYLLABUS

## PHARMACEUTICAL ANALYSIS

### Course of study for M. Pharm. (Pharmaceutical Analysis)

<table>
<thead>
<tr>
<th>Course Code</th>
<th>Course</th>
<th>Credit Hours</th>
<th>Credit Points</th>
<th>Hrs./wk</th>
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<tbody>
<tr>
<td>Semester I</td>
<td></td>
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<tr>
<td>MPT1011</td>
<td>Modern Pharmaceutical Analytical Techniques</td>
<td>4</td>
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<tr>
<td>MPT1012</td>
<td>Advanced Pharmaceutical Analysis</td>
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<td>MPT1013</td>
<td>Pharmaceutical Validation</td>
<td>4</td>
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<td>MPT1014</td>
<td>Food Analysis</td>
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<tr>
<td>MPT1915</td>
<td>Pharmaceutical Analysis Practical - I</td>
<td>12</td>
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<td>MPT1916</td>
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<td>Semester II</td>
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<td>MPT2011</td>
<td>Advanced Instrumental Analysis</td>
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<td>MPT2012</td>
<td>Modern Bio-Analytical Techniques</td>
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<td>MPT2013</td>
<td>Quality Control and Quality Assurance</td>
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<td>MPT2014</td>
<td>Herbal and Cosmetic Analysis</td>
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<td>MPT2915</td>
<td>Pharmaceutical Analysis Practical - II</td>
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Scope
This subject deals with various advanced analytical instrumental techniques for identification, characterization
and quantification of drugs. Instruments dealt are NMR, Mass spectrometer, IR, HPLC, GC etc.

Objectives
After completion of course student is able to know about chemicals and excipients
• The analysis of various drugs in single and combination dosage forms
• Theoretical and practical skills of the instruments

THEORY 60 Hrs
   b. IR spectroscopy: Theory, Modes of Molecular vibrations, Sample handling, Instrumentation of Dispersive and Fourier Transform IR Spectrometer, Factors affecting vibrational frequencies and Applications of IR spectroscopy, Data Interpretation.
   c. Spectrofluorimetry: Theory of Fluorescence, Factors affecting fluorescence (Characteristics of drugs that can be analysed by fluorimetry), Quenchers, Instrumentation and Applications of fluorescence spectrophotometer.
   10 Hrs
2. NMR spectroscopy: Quantum numbers and their role in NMR, Principle, Instrumentation, Solvent requirement in NMR, Relaxation process, NMR signals in various compounds, Chemical shift, Factors influencing chemical shift, Spin-Spin coupling, Coupling constant, Nuclear magnetic double resonance, Brief outline of principles of FT-NMR and 13C NMR. Applications of NMR spectroscopy.
   10 Hrs
   10 Hrs
4. Chromatography: Principle, apparatus, instrumentation, chromatographic parameters, factors affecting resolution, isolation of drug from excipients, data interpretation and applications of the following:
   a. Thin Layer chromatography
   b. High Performance Thin Layer Chromatography
   c. Ion exchange chromatography
   d. Column chromatography
   e. Gas chromatography
   f. High Performance Liquid chromatography
   g. Ultra High Performance Liquid chromatography
   h. Affinity chromatography
   i. Gel Chromatography
   10 Hrs
5. a. Electrophoresis: Principle, Instrumentation, Working conditions, factors affecting separation and applications of the following:
   a) Paper electrophoresis b) Gel electrophoresis c) Capillary electrophoresis d) Zone electrophoresis e) Moving boundary electrophoresis f) Isoelectric focusing
   b. X ray Crystallography: Production of X rays, Different X ray methods, Bragg's law, Rotating crystal technique, X ray powder technique, Types of crystals and applications of X-ray diffraction
   10 Hrs

**Thermal Techniques**: Principle, thermal transitions and Instrumentation (Heat flux and power-compensation and designs), Modulated DSC, Hyper DSC, experimental parameters (sample preparation, experimental conditions, calibration, heating and cooling rates, resolution, source of errors) and their influence, advantage and disadvantages, pharmaceutical applications.

**Differential Thermal Analysis (DTA)**: Principle, instrumentation and advantage and disadvantages, pharmaceutical applications, derivative differential thermal analysis (DDTA).

**TGA**: Principle, instrumentation, factors affecting results, advantage and disadvantages, pharmaceutical applications.

**REFERENCES**

ADVANCED PHARMACEUTICAL ANALYSIS
(MPT 1012)

Scope
This subject deals with the various aspects of Impurity, Impurities in new drug products, in residual solvents, Elemental impurities, Impurity profiling and characterization of degradents, Stability testing of phytopharmaceuticals and their protocol preparation. It also covers the biological testing of various vaccines and their principle and procedure.

Objective
After completion of the course students shall able to know,

- Appropriate analytical skills required for the analytical method development.
- Principles of various reagents used in functional group analysis that renders necessary support in research methodology and demonstrates its application in the practical related problems.
- Analysis of impurities in drugs, residual solvents and stability studies of drugs and biological products

THEORY

1. Impurity and stability studies:
   Definition, classification of impurities in drug Substance or Active Pharmaceutical Ingredients and quantification of impurities as per ICH guidelines
   Rationale for the reporting and control of degradation products, reporting degradation products content of batches, listing of degradation products in specifications, qualification of degradation products
   Impurities in residual solvents:
   General principles, classification of residual solvents, Analytical procedures, limits of residual solvents, reporting levels of residual solvents

2. Elemental impurities:
   Element classification, control of elemental impurities, Potential Sources of elemental Impurities, Identification of Potential Elemental Impurities, analytical procedures, instrumentation & C, H, N and S analysis
   Stability testing protocols:
   Selection of batches, container orientation, test parameters, sampling frequency, specification, storage conditions, recording of results, concept of stability, commitment etc.
   Important mechanistic and stability related information provided by results of study of factors like temperature, pH, buffering species, ionic strength and dielectric constant etc. on the reaction rates. With practical considerations

3. Impurity profiling and degradent characterization:
   Method development, Stability studies and concepts of validation accelerated stability testing & shelf life calculation, WHO and ICH stability testing guidelines, Stability zones, steps in development, practical considerations. Basics of impurity profiling and degradent characterization with special emphasis. Photostability testing guidelines, ICH stability guidelines for biological products

4. Stability testing of phytopharmaceuticals:
   Regulatory requirements, protocols, HPTLC/HPLC finger printing, interactions and complexity.

5. Biological tests and assays of the following:

6. Immunoassays (IA)
   Basic principles, Production of antibodies, Separation of bound and unbound drug, Radioimmunoassay, Optical IA, Enzyme IA, Fluoro IA, Luminiscence IA, Quantification and applications of IA.

REFERENCES

9. Methods of sampling and microbiological examination of water, first revision, BIS
14. ICH Guidelines for impurity profiles and stability studies.
PHARMACEUTICAL VALIDATION  
(MPT 1013)

**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.

**Objectives**
Upon completion of the subject student shall be able to
- Explain the aspect of validation
- Carry out validation of manufacturing processes
- Apply the knowledge of validation to instruments and equipments
- Validate the manufacturing facilities

**THEORY**

<table>
<thead>
<tr>
<th>THEORETICAL TOPICS</th>
<th>HOURS</th>
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<tbody>
<tr>
<td>Introduction: Definition of Qualification and Validation, Advantage of Validation, Streamlining of Qualification &amp; Validation process and Validation Master Plan, Qualification; User Requirement Specification, Design Qualification, Factory Acceptance Test (FAT) Site Acceptance Test (SAT), Installation Qualification, Operational Qualification, Performance Qualification, Re-Qualification (Maintaining status- Calibration Preventive Maintenance, Change management), Qualification of Manufacturing Equipments, Qualification of Analytical Instruments and Laboratory equipments.</td>
<td>12 Hrs</td>
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<tr>
<td>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.</td>
<td>12 Hrs</td>
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<tr>
<td>Analytical method validation: General principles, Validation of analytical method as per ICH guidelines and USP, Computerized system validation: Electronic records and digital significance-21CFR part11and GAMP 5.</td>
<td>12 Hrs</td>
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<tr>
<td>General Principles of Intellectual Property: Concepts of Intellectual Property (IP), Intellectual Property Protection (IPP), Intellectual Property Rights (IPR); Economic importance, mechanism for protection of Intellectual Property - patents, Copyright, Trademark; Factors affecting choice of IP protection; Penalties for violation; Role of IP in pharmaceutical industry; Global ramification and financial implications. Filing a patent applications; patent application forms and guidelines. Types patent applications-provisional and non-provisional, PCT and convention patent applications; International patenting requirement procedures and costs; Rights and responsibilities of a patentee; Practical aspects regarding maintaining of a Patent file; Patent infringement meaning and scope. Significance of transfer technology (TOT), IP and ethics-positive and negative aspects of IPP; Societal responsibility, avoiding unethical practices.</td>
<td>12 Hrs</td>
</tr>
</tbody>
</table>

**REFERENCES**

3. Validation Masterplan by Terveeks or Deeks, Davis Harwood International publishing.
7. Pharmaceutical Equipment Validation: The Ultimate Qualification Handbook, Phillip A. Cloud,
Interpharm Press


FOOD ANALYSIS
(MPT 1014)

Scope
This course is designed to impart knowledge on analysis of food constituents and finished food products. The course includes application of instrumental analysis in the determination of pesticides in variety of food products.

Objectives
At completion of this course student shall be able to understand various analytical techniques in the determination of:
- Food constituents
- Food additives
- Finished food products
- Pesticides in food
- And also student shall have the knowledge on food regulations and legislations

THEORY 60 Hrs


5. Pesticide analysis: Effects of pest and insects on various food, use of pesticides in agriculture, pesticide cycle, organophosphorus and organochlorine pesticides analysis, determination of pesticide residues in grain, fruits, vegetables, milk and milk products. Legislation regulations of food products with special emphasis on BIS, Agmark, FDA and US-FDA.

REFERENCES

4. Analysis of Food constituents - Multon, Wiley VCH.
1. Analysis of Pharmacopoeial compounds and their formulations by UV Vis spectrophotometer
2. Simultaneous estimation of multicomponent containing formulations by UV spectrophotometry
3. Experiments based on HPLC
4. Experiments based on Gas Chromatography
5. Estimation of riboflavin/quinine sulphate by fluorimetry
6. Estimation of sodium/potassium by flame photometry
7. Assay of official compounds by different titrations
8. Assay of official compounds by instrumental techniques.
9. Quantitative determination of hydroxyl group.
10. Quantitative determination of amino group
11. Colorimetric determination of drugs by using different reagents
12. Impurity profiling of drugs
13. Calibration of glassware
14. Calibration of pH meter
15. Calibration of UV-Visible spectrophotometer
16. Calibration of FTIR spectrophotometer
17. Calibration of GC instrument
18. Calibration of HPLC instrument
19. Cleaning validation of any one equipment
20. Determination of total reducing sugar
21. Determination of proteins
22. Determination of saponification value, Iodine value, Peroxide value, Acid value in food products
23. Determination of fat content and rancidity in food products
24. Analysis of natural and synthetic colors in food
25. Determination of preservatives in food
26. Determination of pesticide residue in food products
27. Analysis of vitamin content in food products
28. Determination of density and specific gravity of foods
29. Determination of food additives
ADVANCED INSTRUMENTAL ANALYSIS
(MPT 2011)

Scope
This subject deals with various hyphenated analytical instrumental techniques for identification, characterization and quantification of drugs. Instruments dealt are LC-MS, GC-MS, and hyphenated techniques.

Objectives
After completion of course student is able to know,
• interpretation of the NMR, Mass and IR spectra of various organic compounds
• theoretical and practical skills of the hyphenated instruments
• identification of organic compounds

THEORY

1. 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 in pharmaceutical analysis. Immobilized polysaccharide CSP’s: Advancement in enantiomeric separations, revised phase Chiral method development and HILIC approaches. HPLC in Chiral analysis of pharmaceuticals. Preparative HPLC, practical aspects of preparative HPLC. 12 Hrs

2. Biochromatography: Size exclusion chromatography, ion exchange chromatography, ion pair chromatography, affinity chromatography general principles, stationary phases and mobile phases. Gas chromatography: Principles, instrumentation, derivatization, head space sampling, columns for GC, detectors, quantification. High performance Thin Layer chromatography: Principles, instrumentation, pharmaceutical applications. 12 Hrs


4. Mass spectrometry: Principle, theory, instrumentation of mass spectrometry, different types of ionization like electron impact, chemical, field, FAB and MALD, APCI, ESI, APPI mass fragmentation and its rules, meta stable ions, isotopic peaks and applications of mass spectrometry. LC-MS hyphenation and DART MS analysis. Mass analysers (Quadrpole, Time of flight, FT-ICR, ion trap and Orbitrap) instruments. MS/MS systems (Tandem: QqQ, TOF-TOF;Q-IT, Q-TOF, LTQ-FT, LTQ-Orbitrap. 12 Hrs

5. NMR spectroscopy: Quantum numbers and their role in NMR, Principle, Instrumentation, Solvent requirement in NMR, Relaxation process, NMR signals in various compounds, Chemical shift, Factors influencing chemical shift, Spin-Spin coupling, Coupling constant, Nuclear magnetic double resonance, Brief outline of principles of FT-NMR with reference to 13C NMR: Spin spin and spin lattice relaxation phenomenon. 13C NMR, 1-D and 2-D NMR, NOESY and COSY techniques, Interpretation and Applications of NMR spectroscopy. LC-NMR hyphenations. 12 Hrs

REFERENCES

New Delhi, 1997.


MODERN BIO-ANALYTICAL TECHNIQUES
(MPT 2012)

Scope
This subject is designed to provide detailed knowledge about the importance of analysis of drugs in biological matrices.

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

- Extraction of drugs from biological samples
- Separation of drugs from biological samples using different techniques
- Guidelines for BA/BE studies.

THEORY 60 Hrs

1. 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. 12 Hrs

2. Biopharmaceutical Consideration:

3. Pharmacokinetics and Toxicokinetics:
   Basic consideration, Drug interaction (PK-PD interactions), The effect of protein-binding interactions, The effect of tissue-binding interactions, Cytochrome P450-based drug interactions, Drug interactions linked to transporters. Microsomal assays Toxicokinetics-Toxicokinetic evaluation in preclinical studies, Importance and applications of toxicokinetic studies. LC-MS in bioactivity screening and proteomics. 12 Hrs

4. Cell culture techniques
   Basic equipments used in cell culture lab. Cell culture media, various types of cell culture, general procedure for cell cultures; isolation of cells, subculture, cryopreservation, characterization of cells and their applications. Principles and applications of cell viability assays (MTT assays), Principles and applications of flow cytometry. 12 Hrs

5. Metabolite identification:

REFERENCES

8. Good Laboratory Practice Regulations, 2ndEdition, Sandy Weinberg Vol. 69, Marcel Dekker Series,
10. ICH, USFDA & CDSCO Guidelines.
11. Palmer
QUALITY CONTROL AND QUALITY ASSURANCE
(MPT 2013)

Scope
This course deals with the various aspects of quality control and quality assurance aspects of pharmaceutical industries. It covers the important aspects like cGMP, QC tests, documentation, quality certifications, GLP and regulatory affairs.

Objectives
At the completion of this subject it is expected that the student shall be able to know

- the cGMP aspects in a pharmaceutical industry
- to appreciate the importance of documentation
- to understand the scope of quality certifications applicable to Pharmaceutical industries
- to understand the responsibilities of QA & QC departments

THEORY 60 Hrs

1. Concept and Evolution of Quality Control and Quality Assurance
   Good Laboratory Practice, GMP, Overview of ICH Guidelines -QSEM, with special emphasis on Q-series guidelines.
   Good Laboratory Practices: Scope of GLP, Definitions, Quality assurance unit, protocol for conduct of non clinical testing, control on animal house, report preparation and documentation.
   12 Hrs

2. cGMP guidelines according to schedule M, USFDA (inclusive of CDER and CBER) Pharmaceutical Inspection Convention (PIC), WHO and EMEA covering: Organization and personnel responsibilities, training, hygiene and personal records, drug industry location, design, construction and plant lay out, maintenance, sanitation, environmental control, utilities and maintenance of sterile areas, control of contamination and Good Warehousing Practice. CPCEA guidelines.
   12 Hrs

3. Analysis of raw materials, finished products, packaging materials, in process quality control (IPQC), Developing specification (ICH Q6 and Q3)
   Purchase specifications and maintenance of stores for raw materials. In process quality control and finished products quality control for following formulation in Pharma industry according to Indian, US and British pharmacopoeias: tablets, capsules, ointments, suppositories, creams, parenterals, ophthalmic and surgical products (How to refer pharmacopoeias), Quality control test for containers, closures and secondary packing materials.
   12 Hrs

4. Documentation in pharmaceutical industry: Three tier documentation, Policy, Procedures and Work instructions, and records (Formats), Basic principles- How to maintain, retention and retrieval etc. Standard operating procedures (How to write), Master Formula Record, Batch Formula Record, Quality audit plan and reports. Specification and test procedures, Protocols and reports. Distribution records. Electronic data.
   12 Hrs

5. Manufacturing operations and controls: Sanitation of manufacturing premises, mix-ups and cross contamination, processing of intermediates and bulk products, packaging operations, IPQC, release of finished product, process deviations, charge-in of components, time limitations on production, drug product inspection, expiry date calculation, calculation of yields, production record review, change control, sterile products, aseptic process control, packaging.
   12 Hrs

REFERENCES

7. ICH guidelines
8. ISO 9000 and total quality management
HERBAL AND COSMETIC ANALYSIS
(MPT 2014)

Scope
This course is designed to impart knowledge on analysis of herbal products. Regulatory requirements, herbal drug interaction with monographs. Performance evaluation of cosmetic products is included for the better understanding of the equipments used in cosmetic industries for the purpose.

Objectives
At completion of this course student shall be able to understand:
- Determination of herbal remedies and regulations
- Analysis of natural products and monographs
- Determination of Herbal drug-drug interaction
- Principles of performance evaluation of cosmetic products.

THEORY 60 Hrs


2. Adulteration and Deterioration: Introduction, types of adulteration/substitution of herbal drugs, Causes and Measure of adulteration, Sampling Procedures, Determination of Foreign Matter, DNA Finger printing techniques in identification of drugs of natural origin, heavy metals, pesticide residues, phototoxin and microbial contamination in herbal formulations. Regulatory requirements for setting herbal drug industry: Global marketing management, Indian and international patent law as applicable herbal drugs and natural products and its protocol. 12 Hrs


4. Herbal drug-drug interaction: WHO and AYUSH guidelines for safety monitoring of natural medicine, Spontaneous reporting schemes for bio drug adverse reactions, bio drug-drug and bio drug-food interactions with suitable examples. Challenges in monitoring the safety of herbal medicines. 12 Hrs

5. Evaluation of cosmetic products: Determination of acid value, ester value, saponification value, iodine value, peroxide value, rancidity, moisture, ash, volatile matter, heavy metals, fineness of powder, density, viscosity of cosmetic raw materials and finished products. Study of quality of raw materials and general methods of analysis of raw material used in cosmetic manufacture as per BIS. Indian Standard specification laid down for sampling and testing of various cosmetics in finished forms such as baby care products, skin care products, dental products, personal hygiene preparations, lips sticks, Hair products and skin creams by the Bureau Indian Standards. 12 Hrs

REFERENCES

1. Pharmacognosy by Trease and Evans
2. Pharmacognosy by Kokate, Purohit and Gokhale
4. Pharmacognosy & Pharmacobiotechnology by AshutoshKar
5. Essential of Pharmacognosy by Dr. S. H. Ansari
8. Indian Standard specification for 28 finished cosmetics BIS, New Delhi
9. Harry's Cosmetology 8th edition
10. Suppliers catalogue on specialized cosmetic excipients
PHARMACEUTICAL ANALYSIS PRACTICALS – I
(MPT 2915)

1. Comparison of absorption spectra by UV and Woodward - Fiesure rule
2. Interpretation of organic compounds by FT-IR
3. Interpretation of organic compounds by NMR
4. Interpretation of organic compounds by MS
5. Determination of purity by DSC in pharmaceuticals
6. Identification of organic compounds using FT-IR, NMR, CNMR and Mass spectra
7. Bio molecules separation utilizing various sample preparation techniques and Quantitative analysis of components by gel electrophoresis.
8. Bio molecules separation utilizing various sample preparation techniques and Quantitative analysis of components by HPLC techniques.
9. Isolation of analgesics from biological fluids (Blood serum and urine).
11. Protocol preparation for the conduct of BA/BE studies according to guidelines.
12. In process and finished product quality control tests for tablets, capsules, parenterals and creams
13. Quality control tests for Primary and secondary packing materials
14. Assay of raw materials as per official monographs
15. Testing of related and foreign substances in drugs and raw materials
16. Preparation of Master Formula Record.
17. Preparation of Batch Manufacturing Record.
18. Quantitative analysis of rancidity in lipsticks and hair oil
19. Determination of aryl amine content and Developer in hair dye
20. Determination of foam height and SLS content of Shampoo.
21. Determination of total fatty matter in creams (Soap, skin and hair creams)
22. Determination of acid value and saponification value.
23. Determination of calcium thioglycolate in depilatories