**Table 5: Course of study for M. Pharm (Pharmaceutical Analysis)**

**Semester I**

| Course Code | Course | Credit Hours | Credit Points | Hrs./wk | Marks |
|---|---|---|---|---|---|
| MPA 101T | Modern Pharmaceutical Analytical Techniques | 4 | 4 | 4 | 100 |
| MPA 102T | Advanced Pharmaceutical Analysis | 4 | 4 | 4 | 100 |
| MPA 103T | Pharmaceutical Validation | 4 | 4 | 4 | 100 |
| MPA 104T | Food Analysis | 4 | 4 | 4 | 100 |
| MPA 105P | Pharmaceutical Analysis Practical I | 12 | 6 | 12 | 150 |
| - | Seminar/Assignment | 7 | 4 | 7 | 100 |
| **Total** | | **35** | **26** | **35** | **650** |

**Semester II**

| Course Code | Course | Credit Hours | Credit Points | Hrs./wk | Marks |
|---|---|---|---|---|---|
| MPA 201T | Advanced Instrumental Analysis | 4 | 4 | 4 | 100 |
| MPA 202T | Modern Bio-Analytical Techniques | 4 | 4 | 4 | 100 |
| MPA 203T | Quality Control and Quality Assurance | 4 | 4 | 4 | 100 |
| MPA 204T | Herbal and Cosmetic Analysis | 4 | 4 | 4 | 100 |
| MPA 205P | Pharmaceutical Analysis Practical II | 12 | 6 | 12 | 150 |
| - | Seminar/Assignment | 7 | 4 | 7 | 100 |
| **Total** | | **35** | **26** | **35** | **650** |

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PHARMACEUTICALANALYSIS(MPA)

MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES
(MPA 101T)

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 |
|---|---|---|
| 1. | a. UV-Visible spectroscopy: Introduction, Theory, Laws, Instrumentation associated with UV-Visible spectroscopy, Choice of solvents and solvent effect and Applications of UV-Visible spectroscopy, Difference/ Derivative spectroscopy.<br>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.<br>c. Spectroflourimetry: Theory of Fluorescence, Factors affecting fluorescence (Characterestics of drugs that can be analysed by flourimetry), Quenchers, Instrumentation and Applications of fluorescence spectrophotometer.<br>d. Flame emission spectroscopy and Atomic absorption spectroscopy: Principle, Instrumentation, Interferences and Applications. | 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 |
| 3 | Mass Spectroscopy: Principle, Theory, Instrumentation of Mass | 10 |

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|---|---|---|
| | Spectroscopy, Different types of ionization like electron impact, chemical, field, FAB and MALDI, APCI, ESI, APPI Analyzers of Quadrupole and Time of Flight, Mass fragmentation and its rules, Meta stable ions, Isotopic peaks and Applications of Mass spectroscopy. | Hrs |
| 4 | Chromatography: Principle, apparatus, instrumentation, chromatographic parameters, factors affecting resolution, isolation of drug from excipients, data interpretation and applications of the following:<br>a. Thin Layer chromatography<br>b. High Performance Thin Layer Chromatography<br>c. Ion exchange chromatography<br>d. Column chromatography<br>e. Gas chromatography<br>f. High Performance Liquid chromatography<br>g. Ultra High Performance Liquid chromatography<br>h. Affinity chromatography<br>i. Gel Chromatography | 10 Hrs |
| 5 | a. Electrophoresis: Principle, Instrumentation, Working conditions, factors affecting separation and applications of the following:<br>a) Paper electrophoresis b) Gel electrophoresis c) Capillary electrophoresis d) Zone electrophoresis e) Moving boundary electrophoresis f) Iso electric focusing<br><br>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 |
| 6 | Potentiometry: Principle, working, Ion selective Electrodes and Application of potentiometry.<br><br>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 | 10 Hrs |

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and advantage and disadvantages, pharmaceutical applications, derivative differential thermal analysis (DDTA). TGA: Principle, instrumentation, factors affecting results, advantage and disadvantages, pharmaceutical applications.

REFERENCES

1. Spectrometric Identification of Organic compounds – Robert M Silverstein, Sixth edition, John Wiley & Sons, 2004.
2. Principles of Instrumental Analysis – Doglas A Skoog, F. James Holler, Timothy A. Nieman, 5th edition, Eastern press, Bangalore, 1998.
3. Instrumental methods of analysis – Willards, 7th edition, CBS publishers.
4. Practical Pharmaceutical Chemistry – Beckett and Stenlake, Vol II, 4th edition, CBS Publishers, New Delhi, 1997.
5. Organic Spectroscopy – William Kemp, 3rd edition, ELBS, 1991.
6. Quantitative Analysis of Drugs in Pharmaceutical formulation – P D Sethi, 3rd Edition, CBS Publishers, New Delhi, 1997.
7. Pharmaceutical Analysis – Modern Methods – Part B – J W Munson, Vol 11, Marcel. Dekker Series
8. Spectroscopy of Organic Compounds, 2nd edn., P.S/Kalsi, Wiley estern Ltd., Delhi.
9. Textbook of Pharmaceutical Analysis, KA.Connors, 3rd Edition, John Wiley & Sons, 1982.

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## ADVANCED PHARMACEUTICAL ANALYSIS
## (MPA 102T)

### 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 | 60 Hrs |
|---|---|---|
| 1. | Impurity and stability studies:<br>Definition, classification of impurities in drug Substance or Active Pharmaceutical Ingredients and quantification of impurities as per ICH guidelines<br>Impurities in new drug products:<br>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<br>Impurities in residual solvents:<br>General principles, classification of residual solvents, Analytical procedures, limits of residual solvents, reporting levels of residual solvents | 10 Hrs |
| 2 | Elemental impurities:<br>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 | 10 Hrs |

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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.

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| 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 | 10 Hrs |
| 4 | Stability testing of phytopharmaceuticals: Regulatory requirements, protocols, HPTLC/HPLC finger printing, interactions and complexity. | 10 Hrs |
| 5 | Biological tests and assays of the following: a. Adsorbed Tetanus vaccine b. Adsorbed Diphtheria vaccine c. Human anti haemophilic vaccine d. Rabies vaccine e. Tetanus Anti toxin f. Tetanus Anti serum g. Oxytocin h. Heparin sodium IP i. Antivenom. PCR, PCR studies for gene regulation, instrumentation (Principle and Procedures) | 10 Hrs |
| 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. | 10 Hrs |

REFERENCES

1. Vogel's textbook of quantitative chemical analysis – Jeffery J Bassett, J. Mendham, R. C. Denney, 5th edition, ELBS, 1991.
2. Practical Pharmaceutical Chemistry – Beckett and Stenlake, Vol II, 4th Edition, CBS publishers, New Delhi, 1997.
3. Textbook of Pharmaceutical Analysis – K A Connors, 3rd Edition, John Wiley & Sons, 1982.

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4. Pharmaceutical Analysis – Higuchi, Brochmman and Hassen, 2nd Edition, Wiley – Inter science Publication, 1961.
5. Quantitative Analysis of Drugs in Pharmaceutical formulation – P D Sethi, 3rd Edition, CBS Publishers New Delhi, 1997.
6. Pharmaceutical Analysis– Modern methods – J W Munson – Part B, Volume 11, Marcel Dekker Series.
7. The Quantitative analysis of Drugs – D C Carratt, 3rd edition, CBS Publishers, NewDelhi, 1964.
8. Indian Pharmacopoeia Vol I , II & III 2007, 2010, 2014.
9. Methods of sampling and microbiological examination of water, first revision, BIS
10. Practical HPLC method development – Snyder, Kirkland, Glajch, 2nd edition, John Wiley & Sons.
11. Analytical Profiles of drug substances – Klaus Florey, Volume 1 – 20, Elsevier, 2005
12. Analytical Profiles of drug substances and Excipients – Harry G Brittan, Volume 21 – 30, Elsevier, 2005.
13. The analysis of drugs in biological fluids – Joseph Chamberlain, 2nd edition, CRC press, London.
14. ICH Guidelines for impurity profiles and stability studies.

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## PHARMACEUTICAL VALIDATION
## (MPA 103T)

### 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
- Carryout validation of manufacturing processes
- Apply the knowledge of validation to instruments and equipments
- Validate the manufacturing facilities

THEORY 60 Hrs

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|---|---|---|
| 1. | Introduction: Definition of Qualification and Validation, Advantage of Validation, Streamlining of Qualification & Validation process and Validation Master Plan.<br>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. | 12 Hrs |
| 2 | Qualification of analytical instruments: Electronic balance, pH meter, UV-Visible spectrophotometer, FTIR, GC, HPLC, HPTLC<br>Qualification of Glassware: Volumetric flask, pipette, Measuring cylinder, beakers and burette. | 12 Hrs |
| 3 | Validation of Utility systems: Pharmaceutical Water System & pure steam, HVAC system, Compressed air and nitrogen.<br>Cleaning Validation: Cleaning Validation - Cleaning Method development, Validation and validation of analytical method used in cleaning. Cleaning of Equipment, Cleaning of Facilities. Cleaning in place (CIP). | 12 Hrs |
| 4 | Analytical method validation: General principles, Validation of analytical method as per ICH guidelines and USP. | 12 Hrs |

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| | Computerized system validation: Electronic records and digital significance-21 CFR part 11 and GAMP 5. | 12 Hrs |
| 5 | 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. | |

REFERENCES

1. B. T. Loftus & R. A. Nash, "Pharmaceutical Process Validation", Drugs and Pharm Sci. Series, Vol. 129, 3rd Ed., Marcel Dekker Inc., N.Y.
2. The Theory & Practice of Industrial Pharmacy, 3rd edition, 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, 2nd Edition, by Carleton & Agalloco, (Marcel Dekker).
5. Michael Levin, Pharmaceutical Process Scale-Up , Drugs and Pharm. Sci. Series, Vol. 157,2nd Ed., Marcel Dekker Inc., N.Y.
6. 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, 2nd Ed.
9. Analytical Method validation and Instrument Performance Verification by Churg Chan, Heiman Lam, Y.C. Lee, Yue. Zhang, Wiley Inter Science.

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## FOOD ANALYSIS
## (MPA 104T)

### 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

1. Carbohydrates: classification and properties of food carbohydrates, General methods of analysis of food carbohydrates, Changes in food carbohydrates during processing, Digestion, absorption and metabolism of carbohydrates, Dietary fibre, Crude fibre and application of food carbohydrates 12 Hrs
   Proteins: Chemistry and classification of amino acids and proteins, Physico-Chemical properties of protein and their structure, general methods of analysis of proteins and amino acids, Digestion, absorption and metabolism of proteins.

2 Lipids: Classification, general methods of analysis, refining of fats and oils; hydrogenation of vegetable oils, Determination of adulteration in fats and oils, Various methods used for measurement of spoilage of fats and fatty foods. 12 Hrs
   Vitamins: classification of vitamins, methods of analysis of vitamins, Principles of microbial assay of vitamins of B-series.

3 Food additives: Introduction, analysis of Preservatives, antioxidants, artificial sweeteners, flavors, flavor enhancers, stabilizers, thickening and jelling agents. 12 Hrs
   Pigments and synthetic dyes: Natural pigments, their occurrence and characteristic properties, permitted synthetic

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dyes, Non-permitted synthetic dyes used by industries, Method of detection of natural, permitted and non-permitted dyes.

| | | |
|---|---|---|
| 4 | General Analytical methods for milk, milk constituents and milk products like ice cream, milk powder, butter, margarine, cheese including adulterants and contaminants of milk.<br>Analysis of fermentation products like wine, spirits, beer and vinegar. | 12 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.<br>Legislation regulations of food products with special emphasis on BIS, Agmark, FDA and US-FDA. | 12 Hrs |

REFERENCES

1. The chemical analysis of foods – David Pearson, Seventh edition, Churchill Livingstone, Edinburgh London, 1976
2. Introduction to the Chemical analysis of foods – S. Nielsen, Jones & Bartlett publishers, Boston London, 1994.
3. Official methods of analysis of AOAC International, sixth edition, Volume I & II, 1997.
4. Analysis of Food constituents – Multon, Wiley VCH.
5. Dr. William Horwitz, Official methods of analysis of AOAC International, 18th edition, 2005.

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PHARMACEUTICAL ANALYSIS PRACTICALS - II
(MPA 105P)

1. Analysis of Pharmacopoeial compounds and their formulations by UV Vis spectrophotometer
2. Simultaneous estimation of multi component 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. Imupurity profiling of drugs
13. Calibration of glasswares
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

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