INSTITUTE OF SCIENCE, TECHNOLOGY & ADVANCED STUDIES (VISTAS)
(Deemed to be University Estd. u/s 3 of the UGC Act, 1956)
PALLAVARAM - CHENNAI
NAAC ACCREDITED WITH 'A' GRADE
Marching Beyond 25 Years Successfully

M.PHARM

[MASTER OF PHARMACY]

PHARMACEUTICAL ANALYSIS

Curriculum and Syllabus

Effective from the Academic Year 2018-2019

School of Pharmaceutical Sciences

SYLLABUS

Syllabus

Master of Pharmacy

Pharmaceutical Analysis

17MPA 101T

MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

Course Objectives: Employability.

After completion of course student is able to know about chemicals and excipients

1. The analysis of various drugs in single and combination dosage forms
2. Theoretical and practical skills of the instruments

At the end of this course the students will be able to,

CO 1: Demonstrate the analytical instrumental techniques for identification, characterization and quantification of drugs by UV-Visible, IR, Fluorometer, Atomic absorption spectrum and Flame emission

CO2: Illustrate the Principle, theory and the analytical techniques for identifications and characterization of compounds by NMR

CO3: Explain the Principle, theory and the analytical techniques for identifications and characterization of compounds by mass spectroscopy

CO4: Explain the principle and types of chromatographic technique with detailed emphasis on instrumentation and applications

UNIT I **10 HRS**

UV-Visible spectroscopy: Introduction, Theory, Laws, Instrumentation associated with UV-Visible spectroscopy 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, choice of solvents and solvent effect and applications of UV-Visible spectroscopy.

Spectroflourimetry: Theory of Fluorescence, Factors affecting fluorescence, Quenchers, Instrumentation and Applications of fluorescence spectroscopy.

Flame emission spectroscopy and Atomic absorption spectroscopy: Principle, Instrumentation, Interferences and Applications.

UNIT II **10 HRS**

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.

UNIT III **10 HRS**

Mass Spectroscopy: Principle, Theory, Instrumentation of Mass Spectroscopy, Different types of ionization techniques like electron impact, chemical, field desorption, FAB and MALDI, APCI, ESI, APPI Analyzers and detectors. Meta stable ions, Isotopic peaks and Applications of Mass spectroscopy

UNIT IV **10 HRS**

Chromatography: Principle, apparatus, instrumentation, chromatographic parameters, factors affecting resolution and applications of the following: a) Paper chromatography b) Thin Layer chromatography c) Ion exchange chromatography d) Column chromatography e) Gas chromatography f) High Performance Liquid chromatography g) Affinity chromatography.

UNIT V **10 HRS**

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) Iso electric focusing 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.

UNIT VI **10 HRS**

Potentiometry: Principle, working, Ion selective Electrodes and Application of potentiometry. 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

TOTAL: 60 HRS

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.

17MPA102T

ADVANCED PHARMACEUTICAL ANALYSIS

Course Objective: (Employability)

1. After completion of the course students shall able to know, Appropriate analytical skills required for the analytical method development.
2. 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
3. **Analysis of impurities in drugs, residual** solvents and stability studies of drugs and biological products

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Examine the various types of impurities present in active Pharmaceutical ingredients, degraded compounds in new drug products and also the impurities present in the solvents its quantification based on ICH guide lines

CO2: Classify the elemental impurities its source, identification and analysis of elements such as nitrogen, hydrogen and sulphur. An overview of stability protocols

CO3: Interpret the impurity profiling of API, new drug products and characterization of degradents and also demonstrate stability test studies

CO4: Establish Stability testing of Phyto-pharmaceuticals and their protocol preparation

CO5: Devise the biological testing of various vaccines and immunoassays and its applications

UNIT I **10 HRS**

Definition, classification of impurities in drug Substance or Active Pharmaceutical Ingredients and quantification of impurities as per ICH guidelines

Impurities in new drug products:

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

UNIT II **10 HRS**

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

UNIT III **10 HRS**

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

UNIT IV **10 HRS**

Stability testing of phytopharmaceuticals:

Regulatory requirements, protocols, HPTLC/HPLC finger printing, interactions and complexity.

UNIT V **10 HRS**

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

UNIT VI **10 HRS**

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.

TOTAL: 60 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.
4. Vogel,,s textbook of quantitative chemical analysis - Jeffery J Bassett, J. Mendham, R. C. Denney, 5th edition, ELBS, 1991.
5. Practical Pharmaceutical Chemistry - Beckett and Stenlake, Vol II, 4th Edition, CBS publishers, New Delhi, 1997.
6. Textbook of Pharmaceutical Analysis - K A Connors, 3rd Edition, John Wiley & Sons,

1982.

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.

17MPA 103T

PHARMACEUTICALVALIDATION

Course Objectives : (Employability)

Upon completion of the subject student shall be able to

1. Explain the aspect of validation
2. Carryout validation of manufacturing processes
3. Apply the knowledge of validation to instruments and equipments
4. Validate the manufacturing facilities

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Explain the concepts of calibration, qualification and validation of analytical equipments

CO2: Illustrate the Qualification of analytical equipments and glass wares

CO3: Develop the concepts validation of utility service and Cleaning validation of equipments employed in the manufacture of pharmaceuticals

C04: Establish analytical method validation of drugs and computer system validation

CO5: Apply Intellectual property rights and know the patent filing procedures

UNIT I **12 HRS**

Introduction to validation: Definition of Qualification and Validation, Advantages of Validation, scope of Validation, Organization for Validation, Validation Master plan, Types of Validation, Streamlining of qualification & 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

UNIT II **12 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.

UNIT III **12 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 analytical method used in cleaning. Cleaning of Equipment,Cleaning of Facilities. Cleaning in place (CIP).

UNIT IV **12 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.

UNIT V **12 HRS**

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**.**

TOTAL: 60 HRS

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Explain the concepts of calibration, qualification and validation of analytical equipments

CO2: Illustrate the Qualification of analytical equipments and glass wares

CO3: Develop the concepts validation of utility service and Cleaning validation of equipments employed in the manufacture of pharmaceuticals

C04: Establish analytical method validation of drugs and *computer system* validation

CO5: Apply Intellectual property rights and know the patent filing procedures

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.

17MPA104T

FOOD ANALYSIS

Course Objectives (Employability)

At completion of this course student shall be able to understand various analytical techniques in the determination of

1. Food constituents
2. Food additives
3. Finished food products
4. Pesticides in food
5. Student shall have the knowledge on food regulations and legislations

12 HRS

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

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.

UNIT II **12 HRS**

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.

Vitamins: classification of vitamins, methods of analysis of vitamins, Principles of microbial assay of vitamins of B-series.

UNIT III **12 HRS**

Food additives: Introduction, analysis of Preservatives, antioxidants, artificial sweeteners, flavors, flavor enhancers, stabilizers, thickening and jelling agents.

Pigments and synthetic dyes: Natural pigments, theiroccurrence and characteristic properties, permitted synthetic dyes, Non-permitted synthetic dyes used by industries, Method of detection of natural, permitted and non-permitted dyes

UNIT IV **12 HRS**

General Analytical methods for milk, milk constituents and milk products like ice cream, milk powder, butter, margarine, cheese including adulterants and contaminants of milk. Analysis of fermentation products like wine, spirits, beer and vinegar..

UNIT V **12 HRS**

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

TOTAL: 60 HRS

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Explain various analytical techniques in the determination of Food constituents such as carbohydrates and proteins its classification, physiochemical properties and general method of analysis of each food constituents

CO2: Explain various analytical techniques in the determination of Food constituents such as Vitamins and lipids, its classification, physiochemical properties and general method of analysis of each food constituents

CO3: Establish various analytical techniques in the determination of Food additives such as preservatives, sweeteners, antioxidants and coloring agents

CO4: Explain various analytical techniques in the determination of finished products such as milk constituents, milk products and fermentation products

CO5: Establish various analytical techniques in the determination of pesticides in food and the regulations of food products with special emphasis on BIS, Agmark, FDA and US-FDA.

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.

17MPA105P

PHARMACEUTICAL ANALYSIS PRACTICALS – I

COURSE OUT COMES (SKILL DEVELOPMENT)

At the end of this course students will be able to,

CO1: Determine the drug /drug combinations by employing instrumental method of analysis

CO2: Evaluate the official compounds by titrimetric methods

CO3: Formulate the calibration of glass wares and analytical equipment"s

CO4: Assess the impurity profile of drugs

CO5: Determine the food constituents such as carbohydrates, proteins and also food additives Preservatives

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 foodsdermination of food additives

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SEMESTER II

17MPA201

ADVANCED INSTRUMENTALANALYSIS

Course Objectives: Employability

After completion of course student is able to know,

1. Interpretation of the NMR, Mass and IR spectra of various organic compounds
2. Theoretical and practical skills of the hyphenated instruments
3. Identification of organic compounds

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Explain the concepts of calibration, qualification and validation of analytical equipments

CO2: Illustrate the Qualification of analytical equipments and glass wares

CO3: Develop the concepts validation of utility service and Cleaning validation of equipments employed in the manufacture of pharmaceuticals

C04: Establish analytical method validation of drugs and computer system validation

CO5: Apply Intellectual property rights and know the patent filing procedures

UNIT I **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 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.

UNIT II **12 HRS**

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.

UNIT III **12 HRS**

Super critical fluid chromatography: Principles, instrumentation, pharmaceutical applications. Capillary electrophoresis: Overview of CE in pharmaceutical analysis, basic configuration, CE characteristics, principles of CE, methods and modes of CE. General considerations and method development in CE, Crown ethers as buffer additives in capillary electrophoresis. CE-MS hyphenation

UNIT IV **12 HRS**

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.

UNIT V **12 HRS**

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 13CNMR: 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

TOTAL: 60 HRS

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. Organic Spectroscopy - William Kemp, 3rd edition, ELBS, 1991.

5. Quantitative analysis of Pharmaceutical formulations by HPTLC - P D Sethi, CBS Publishers, New Delhi.

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, Volume 11, Marcel Dekker Series.Organic Spectroscopy by Donald L. Paviya, 5th Edition

17MPA202T

MODERN BIO-ANALYTICAL TECHNIQUES

Course Objectives: Employability

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

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

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Illustrate extraction of various drugs andmetabolites from biological matrices and establish bio-analytical method validation based on USFDA and EMEA guidelines

CO2: Explain Bioavailability and factors affecting drug bioavailability, invitro dissolution studies. drug release testing models and permeation studies

CO3: Explain Drug interaction, effect of protein-binding interactions, enyme interactions, toxokinetics interactions and bioactivity screening of proteomics

CO4: Prepare the Cell culture media, isolate the cells and explore the characterisation of cells and applications

CO5: Illustrate invivo and invitro approaches of analysis of metabolites & drug metabolizing enzymes

UNIT I **12 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

UNIT II **12 HRS**

Biopharmaceutical Consideration: Introduction, Biopharmaceutical Factors Affecting Drug Bioavailability, In Vitro: Dissolution and Drug Release Testing, Alternative Methods of Dissolution Testing Transport models, Biopharmaceutics Classification System. Solubility: Experimental methods. Permeability: In-vitro, in-situ and In-vivo methods.

UNIT III **12 HRS**

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

UNIT IV **12 HRS**

Cell culture techniques Basic equipment‟s 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.

UNIT V **12 HRS**

Metabolite identification: In-vitro / in-vivo approaches, protocols and sample preparation. Microsomal approaches (Rat liver microsomes (RLM) and Human liver microsomes (HLM) in Met –ID. Regulatory perspectives. In-vitro assay of drug metabolites & drug metabolizing enzymes Drug Product Performance, In Vivo: Bioavailability and Bioequivalence: Drug Product Performance, Purpose of Bioavailability Studies, Relative and Absolute Availability. Methods for Assessing Bioavailability, Bioequivalence Studies, Design and Evaluation of Bioequivalence Studies, Study Designs, Crossover Study Designs, Generic Biologics (Biosimilar Drug Products), Clinical Significance of Bioequivalence Studies.

TOTAL: 60 HRS

REFERENCES

1. Analysis of drugs in Biological fluids - Joseph Chamberlain, 2nd Edition. CRC Press, Newyork. 1995.
2. Principles of Instrumental Analysis - Doglas A Skoog, F. James Holler, Timothy A. Nieman, 5th edition, Eastern press, Bangalore, 1998.
3. Pharmaceutical Analysis - Higuchi, Brochmman and Hassen, 2nd Edition, Wiley – Interscience Publications, 1961.
4. Pharmaceutical Analysis- Modern methods – Part B - J W Munson, Volume 11, Marcel Dekker Series
5. Practical HPLC method Development – Snyder, Kirkland, Glaich, 2nd Edition, John Wiley & Sons, New Jercy. USA.

6 Chromatographic Analysis of Pharmaceuticals – John A Adamovics, 2nd Edition, Marcel Dekker, Newyork, USA. 1997.
7. Chromatographic methods in clinical chemistry & Toxicology – Roger L Bertholf, Ruth E Winecker, John Wiley & Sons, New Jercy, USA. 2007.
8 Good Laboratory Practice Regulations, 2nd Edition, Sandy Weinberg Vol. 69, Marcel Dekker Series, 1995.
9. Good laboratory Practice Regulations – Allen F. Hirsch, Volume 38, Marcel Dekker Series, 1989.ICH, USFDA & CD.

17MPA 203T

QUALITY CONTROL AND QUALITY ASSURANCE

Course Objectives: (Employability)

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

1. The cGMP aspects in a pharmaceutical industry
2. To appreciate the importance of documentation
3. To understand the responsibilities of QA & QC departments
4. To understand the scope of quality certifications applicable to Pharmaceutical industries

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Illustrate the functions and responsibilities of Quality Assurance and Quality Control department

CO2: Explain the cGmps in pharmaceutical industry and guide lines followed by the CPCSEA

CO3: Explain the quality checks for the raw material, finished preparations and packing materials

CO4: Illustrate the documentations in the pharma industry its maintenance and how documents can be retrieved

CO5: Discuss the manufacturing operations, control of manufacturing operations, yield calculations

UNIT I **12 HRS**

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

UNIT II **12 HRS**

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. CPCSEA guidelines

UNIT III **12 HRS**

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.

UNIT IV **12 HRS**

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 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.

UNIT V **12 HRS**

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

TOTAL: 60 HRS

REFERENCES:

1. Quality Assurance Guide by organization of Pharmaceutical Procedures of India, 3rd revised edition, Volume I & II, Mumbai, 1996.
2. Good Laboratory Practice Regulations, 2nd Edition, Sandy Weinberg Vol. 69, Marcel Dekker Series, 1995.
3. Quality Assurance of Pharmaceuticals- A compendium of Guide lines and Related materials Vol I & II, 2nd edition, WHO Publications, 1999.
4. How to Practice GMP‟s – P P Sharma, Vandana Publications, Agra, 1991.

5. The International Pharmacopoeia – vol I, II, III, IV & V - General Methodsof Analysis and Quality specification for Pharmaceutical Substances, Excepients and Dosage forms, 3rd edition, WHO, Geneva, 2005.
6. Good laboratory Practice Regulations – Allen F. Hirsch, Volume 38, MarcelDekker Series, 1989.
7. ICH guidelines
8. ISO 9000 and total quality management
9. The drugs and cosmetics act 1940 – Deshpande, Nilesh Gandhi, 4th edition, Susmit Publishers, 2006.
10. QA Manual – D.H. Shah, 1st edition, Business Horizons, 2000.
11. Good Manufacturing Practices for Pharmaceuticals a plan for total quality control – Sidney H. Willig, Vol. 52, 3rd edition, Marcel Dekker Series.
12. Steinborn L. GMP/ISO Quality Audit Manual for Healthcare Manufacturers and Their Suppliers, Sixth Edition, (Volume 1 - With Checklists and Software Package). Taylor & Francis; 2003.
13. Sarker DK. Quality Systems and Controls for Pharmaceuticals. John Wiley& Sons; 2008.

17MPA 204T

HERBAL AND COSMETIC ANALYSIS

Course Objectives(Employability)

At completion of this course student shall be able to understand

1. Determination of herbal remedies and regulations
2. Analysis of natural products and monographs
3. Determination of Herbal drug-drug interaction
4. Principles of performance evaluation of cosmetic products

COURSE OUTCOME:

At the end of this course students will be able to,

CO1: Explain herbal medicines and validation of herbal medicines, herbal therapies and pharmacokinetic and pharmacodynamics issues of herbal drugs

CO2: Explain adulteration, types of adulteration, determination of adulterants and finger printing

CO3: Elaborate the testing of natural product by modern analytical methods, and development of stability testing protocols. Study of monographs of herbal products

CO4: Illustrate drug –drug interaction, drug food interaction, guidelines for safety monitoring natural medicine by WHO and Ayush

CO5: Analyze the raw materials for cosmetic preparations according to the BUREA OF INDIAN STANDARDS

UNIT I **12 HRS**

Herbal remedies- Toxicity and Regulations: Herbals vs Conventional drugs, Efficacy of herbal medicine products, Validation of Herbal Therapies, Pharmacodynamics and Pharmacokinetic issues. Herbal drug standardization: WHO and AYUSH guidelines

UNIT II **12 HRS**

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

UNIT III **12 HRS**

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.

Testing of natural products and drugs: Effect of herbal medicine on clinical laboratory testing, Adulterant Screening using modern analytical instruments, Regulation and dispensing of herbal drugs, Stability testing of natural products, protocol.

Monographs of Herbal drugs: Study of monographs of herbal drugs and comparative study in IP, USP, Ayurvedic

Pharmacopoeia, American herbal Pharmacopoeia, British herbal Pharmacopoeia, Siddha and Unani Pharmacopoeia, WHO guidelines in quality assessment of herbal drugs

UNIT IV **12 HRS**

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.

UNIT V **12 HRS**

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.

TOTAL: 60 HRS

REFERENCES:

1. Pharmacognosy by Trease and Evans
2. Pharmacognosy by Kokate, Purohit and Gokhale
3. Quality Control Methods for Medicinal Plant, WHO, Geneva
4. Pharmacognosy & Pharmacobiotechnology by As Ashutosh Kar
5. Cosmetics – Formulation, Manufacturing and Quality Control, P.P. Sharma, 4th edition, Vandana Publications Pvt. Ltd., Delhi
6. Indian Standard specification, for raw materials, BIS, New Delhi.
7. Indian Standard specification for 28 finished cosmetics BIS, New Delhi
8. Harry‟s Cosmeticology 8th edition

9. Suppliers catalogue on specialized cosmetic excipients
10. Wilkinson, Moore, seventh edition, George Godwin. Poucher‟s Perfumes, Cosmetics and Soaps
11. Hilda Butler, 10th Edition, Kluwer Academic Publishers. Handbook of Cosmetic Science and Technology, 3rd Edition

17MPA205P

PHARMACEUTICAL ANALYSIS PRACTICALS – II

List of Experiments; (Skill Development)

1. Comparison of absorption spectra by UV and Wood ward – 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).
10. Protocol preparation and performance of analytical/Bioanalytical method validation.
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 total fatty matter in creams (Soap, skin and hair creams)
21. Determination of acid value and saponification value.
22. Determination of calcium thioglycolate in depilatories

COURSE OUT COME:

At the end of this course students will be able to,

Semester III

17MRM301T

Research Methodology & Biostatistics

UNIT – I **12 HRS**

General Research Methodology: Research, objective, requirements, practical difficulties, review of literature, study design, types of studies, strategies to eliminate errors/bias, controls, randomization, crossover design, placebo, blinding techniques

UNIT – II **12 HRS**

Biostatistics: Definition, application, sample size, importance of sample size, factors influencing sample size, dropouts, statistical tests of significance, type of significance tests, parametric tests(students "t" test, ANOVA, Correlation coefficient, regression), non-parametric tests (wilcoxan rank tests, analysis of variance, correlation, chi square test), null hypothesis, P values, degree of freedom, interpretation of P values

UNIT – III **12 HRS**

Medical Research: History, values in medical ethics, autonomy, beneficence, non-maleficence, double effect, conflicts between autonomy and beneficence/non-maleficence, euthanasia, informed consent, confidentiality, criticisms of orthodox medical ethics, importance of communication, control resolution, guidelines, ethics committees, cultural concerns, truth telling, online business practices, conflicts of interest, referral, vendor relationships, treatment of family members, sexual relationships, fatality.

UNIT – IV **12 HRS**

CPCSEA guidelines for laboratory animal facility: Goals, veterinary care, quarantine, surveillance, diagnosis, treatment and control of disease, personal hygiene, location of animal facilities to laboratories, anesthesia, euthanasia, physical facilities, environment, animal husbandry, record keeping, SOPs, personnel and training, transport of lab animals.

UNIT – V **12 HRS**

Declaration of Helsinki: History, introduction, basic principles for all medical research, and additional principles for medical research combined with medical care.

17SMPA 11/ 17SMPA 21

Seminar/ Assignment. & Assignment: (Employability)

Every candidate shall submit a word processed assignment for individual theory courses in their first and second semester specialization which would be evaluated for a maximum of 25 marks with 50% weightage. Seminar: Every candidate shall present on the submitted assignment topic for individual theory courses in their first and second semester specialization which would be evaluated for a maximum of 25 marks with 50% weightage.

Journal Club - 17MPA 302/17MPA 402

17 MPA302 & 17 MPA 402 Every student shall critically appraise the research article of their specialization published in reputed journals. Students are trained for inquiry based learning and critical thinking skills. Students shall access journals adopting search engines and made to collect relevant data, analyze and comment on the findings with the submission of the document evidence and present on the same for assessment.

17MRW 301/17MRW 401 Dissertation/Research Work

Every candidate shall carry out work on an assigned research project under the guidance of a recognized Postgraduate teacher in the third and fourth semester, the result of which shall be written up and submitted in the form of a dissertation. Work for writing the dissertation is aimed at contributing to the development of a spirit of enquiry, besides exposing the candidate to the techniques of research, critical analysis, acquaintance with the latest advances in medical science and the manner of identifying and consulting available literature. The dissertation shall be examined by a minimum of two examiners; one internal (Mentor) and one external examiner (outside the University).