Nirma University

Institute of Pharmacy

Teaching & Examination Scheme of (M. Pharm. - Pharmaceutical Analysis)

Semester I

Sr. No.	Course Code	Course Title	Teaching Scheme				Examination Scheme				
			L	LPW/ PW	T	C	Duration		Component Weightage		
							SEE	LPW/ PW	CE	LPW/ PW	SEE
1	MPA101T	Modern Pharmaceutical Analytical Techniques	4	-	-	4	3.0	-	0.60	-	0.40
2	MPA102T	Advanced Pharmaceutical Analysis	4	-	-	4	3.0	-	0.60	-	0.40
3	MPA103T	Pharmaceutical Validation	4	-	-	4	3.0	-	0.60	-	0.40
4	MPA104T	Food Analysis	4	-	-	4	3.0	-	0.60	-	0.40
5	MPA105P	Pharmaceutical Analysis Practical I	-	12	-	6	-	6.0	-	1.00	-
6	MPA106S	Seminar/Assignment -I	-	7	-	4	-	-	-	1.00	-
		Total	**16**	**19**	**-**	**26**	-	-	-	-	-
			35								

L: Lectures, P/T: Practicals/Tutorial, C: Credits
LPW/PW: Laboratory / Project Work
SEE: Semester End Examination
CE: Continuous Evaluation

NIRMA UNIVERSITY
Institute of Pharmacy

(M.Pharm. - Pharmaceutical Analysis)
(Semester – I)

L	T	P	C
4	-	-	4

Course Code	**MPA101T**
Course Title	**Modern Pharmaceutical Analytical Techniques**

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

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

Course Learning Outcomes (CLO):

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

1 Recall the fundamental theory of different spectroscopic techniques.
2 Recognize the fundamentals, instrumentation and applications of various chromatographic methods S-17
3 Discuss the instrumentation and application of various spectroscopic techniques
4 Describe various electrophoresis and X-ray methods
5 Apply the knowledge of various thermal and electro analytical methods in analysis of drugs and excipients

Syllabus: **Teaching hours**: **60 Hours**

UNIT-I **10 Hours**

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.
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.

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

Flame emission spectroscopy and Atomic absorption spectroscopy:

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

UNIT – II **10 Hours**

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 ^{13}C NMR. Applications of NMR spectroscopy.

UNIT – III **10 Hours**

Mass Spectroscopy: Principle, Theory, Instrumentation of Mass 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.

UNIT – IV **10 Hours**

Chromatography: Principle, apparatus, instrumentation, chromatographic parameters, factors affecting resolution, isolation of drug from excipients, data interpretation and applications of the following:

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

UNIT – V **10 Hours**

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

- Paper electrophoresis
- Gel electrophoresis
- Capillary electrophoresis
- Zone electrophoresis
- Moving boundary electrophoresis
- 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 Hours**

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.

Suggested Readings^: (Latest edition)

1. Silverstein, R. M., Webster, F. X., Kiemle, D. J., & Bryce, D. L.. Spectrometric Identification of Organic Compounds. Johnwiley & sons.
2. Skoog, D. A. H., James, F., & Nieman, T. A. Principles of Instrumental Analysis. Eastern press.
3. Hobart, W. H., Merritt LL, Dean John. A., Instrumental Methods of Analysis. CBS publishers.
4. Beckett, A. H., & Stenlake, J. B. (Eds.). Practical Pharmaceutical Chemistry: Part II Fourth Edition (Vol. 2). A&C Black.
5. Kemp, W. Organic Spectroscopy. ELBS.
6. Shethi, P. D. Quantitative Analysis of Drugs in Pharmaceutical Formulations. CBS Publishers.
7. Munson, J. W. Pharmaceutical Analysis: Modern Methods (Vol. 11). CRC Press.
8. Kalsi, P. S. Spectroscopy of Organic Compounds. Wiley Estern Ltd.
9. Connors, K. A. A Textbook of Pharmaceutical Analysis. NJ: Johnwiley and sons.

L= Lecture, T= Tutorial, P= Practical, C= Credit

^ this is not an exhaustive list

(M.Pharm. - Pharmaceutical Analysis)
(Semester – I)

L	T	P	C
4	-	-	4

Course Code	**MPA102T**
Course Title	**Advanced Pharmaceutical Analysis**

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,

1. 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 Learning Outcomes (CLO):

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

1. Define the impurities and classification of the impurities E-1
2. Recognize the basic principle, preparation of antibodies and applications of immunoassays E-1
3. Understand stability aspects for drug substances and drug products. ENT-13
4. Describe the analytical method development and validation for stability testing as per regulatory guidelines E-1
5. Discuss stability testing for phytopharmaceuticals S-17
6. Explain the analysis of various vaccines and biological products. E-1

Syllabus: **Teaching hours**: **60 Hours**

UNIT – I **10 Hours**

Impurity and stability studies: 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 Hours**

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 Hours**

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 Hours**

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

UNIT – V E-1 **10 Hours**

Biological tests and assays of the following:

- Adsorbed Tetanus vaccine
- Adsorbed Diphtheria vaccine
- Human anti haemophilic vaccine
- Rabies vaccine
- Tetanus Anti toxin
- Tetanus Anti serum
- Oxytocin
- Heparin sodium IP
- Antivenom.
- PCR, PCR studies for gene regulation, instrumentation (Principle and Procedures)

UNIT – VI **10 Hours**

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.

Suggested Readings^: (Latest edition)

1. Mendham, J. Vogels Textbook of Quantitative Chemical Analysis. Pearson Education India.
2. Beckett, A. H., & Stenlake, J. B. (Eds.). Practical Pharmaceutical Chemistry: Part II Fourth Edition (Vol. 2). A&C Black.
3. Connors, K. A. A Textbook of Pharmaceutical Analysis. NJ: Johnwiley and sons.
4. Higuchi, T., Bodin, J. I., & Brochmann-Hanssen, E. Pharmaceutical Analysis. Interscience Publishers.
5. Shethi, P. D. Quantitative Analysis of Drugs in Pharmaceutical Formulations. CBS Publishers.
6. Munson, J. W. Pharmaceutical Analysis: Modern Methods (Vol. 11). CRC Press.
7. Carratt, D. C. The Quantitative Analysis of Drugs. CBS Publishers.
8. Indian Pharmacopoeia,. Government of India. Ministry of health and family welfare.
9. Methods of Sampling and Microbiological Examination of Water, First Revision, BIS.
10. Snyder, L. R., Kirkland, J. J., & Glajch, J. L. Practical HPLC Method Development. John Wiley & Sons.
11. O. Brien, M., McCauley, J., & Cohen, E. Analytical Profile of Drug Substances, Klaus Florey.
12. Brittain, H. G. Analytical Profiles of Drug Substances and Excipients (Vol. 23). Academic Press.
13. Chamberlain, J. The Analysis of Drugs in Biological Fluids 2nd Edition. CRC press.
14. ICH Guidelines for impurity profiles and stability studies

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(M.Pharm. - Pharmaceutical Analysis)
(Semester – I)

L	T	P	C
4	-	-	4

Course Code	**MPA103T**
Course Title	**Pharmaceutical Validation**

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

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 Learning Outcomes (CLO):

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

1. Define qualification for the analytical instruments and laboratory equipments E-1
2. Understand different types of validation ENT-13
3. Explain water and HVAC system in pharmaceutical industry. E-1
4. Describe the analytical method development and validation for drug substance and drug product as per regulatory guidelines S-19
5. Discuss IPR issues, patent filing, copyright and trademarks ENT-13

Syllabus: **Teaching hours**: **60 Hours**

UNIT – I E **12 Hours**

Introduction: Definition of Qualification and Validation, Advantage 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 ENT 12 Hours

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 E 12 Hours

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 S 12 Hours

Analytical method validation: General principles, Validation of analytical method as per ICH guidelines and USP.

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

UNIT - V ENT 12 Hours

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.

Suggested Readings^: (Latest edition)

1. Loftus, B. T., & Nash, R. A. Pharmaceutical Process Validation. Drugs and Pharm Sci. Series (Vol. 129) M. Dekker.
2. Lachman, L., Lieberman, H. A., & Kanig, J. L. The Theory and Practice of Industrial Pharmacy. Philadelphia: Lea & Febiger. Varghese Publishing House.
3. Terveeks, & Deeks. Validation Master Plan. Davis Harwood International Publishing.
4. Carleton, F. J., & Agalloco, J. P. Validation of Aseptic Pharmaceutical Processes, Marcel Dekker.
5. Levin Michael, Pharmaceutical Process Scale-Up II, Drugs and Pharm. Sci. Series (Vol. 157). Marcel Dekker Inc.
6. Validation Standard Operating Procedures, Step by step guide for achieving compliance in the pharmaceutical, Medical device and biotech industries. Syed Imtiaz Haider. CRC Press.
7. Cloud, P. Pharmaceutical Equipment Validation: The Ultimate Qualification Guidebook. CRC Press.

8. Carleton, F. J., & Agalloco, J. P. Validation of Pharmaceutical Processes: Sterile Products. Informa Healthcare.
9. Chan, C. C., Lee, Y. C., Lam, H., & Zhang, X. M. (Eds.). Analytical Method Validation and Instrument Performance Verification. John Wiley & Sons.

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(M.Pharm. - Pharmaceutical Analysis)
(Semester – I)

L	T	P	C
4	-	-	4

Course Code	**MPA104T**
Course Title	**Food Analysis**

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

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

Course Learning Outcomes (CLO):

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

1. Recall and classify different carbohydrate and protein E-1
2. Discuss different types of lipid and vitamins along with general method of analysis S-17
3. Describe fundamentals of standards and quality for food products and additives. ENT-13
4. Tell food legislation E-1
5. Apply different analytical methods for dairy products and beverages ENT-13
6. Use analytical methods for the determination of pesticides. S-19

Syllabus: **Teaching hours**: **60 Hours**

UNIT – I **12 Hours**

Carbohydrates: Chemistry & 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 Hours**

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 and physiological significance of vitamins of B-series.

UNIT – III **12 Hours**

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

Pigments and synthetic dyes: Natural pigments, their occurrence 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 ENT-13 **12 Hours**

General Analytical methods: 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 E-1, S-19 **12 Hours**

Pesticide analysis: Effects of pest and insects on various food, use of pesticides in agriculture, pesticide cycle, organophosphorous and organo chlorine 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.

Suggested Readings^: (Latest edition)

1. Pearson, D. The Chemical Analysis of Foods Longman Group Ltd.
2. Nielsen, S. S. (Ed.). Introduction to the Chemical Analysis of Foods. Sudbury, MA: Jones and Bartlett.
3. Cuniff, P. Official Methods of Analysis of AOAC International. AOAC International.
4. Multon, J. L. Analysis of Food Constituents. John Wiley & Sons.
5. Horwitz, W. Official Methods of Analysis of the AOAC International. The Association.

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(M.Pharm. - Pharmaceutical Analysis)
(Semester – I)

L	T	P	C
-	-	12	6

Course Code	MPA105P
Course Title	Pharmaceutical Analysis Practical I

Syllabus: **Teaching hours**: **180 Hours**

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. Impurity 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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