Nirma University
Institute of Pharmacy
Teaching & Examination Scheme of (M. Pharm. - Pharmaceutical Analysis)
Semester II

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	MPA201T	Advanced Instrumental Analysis	4	-	-	4	3.0	-	0.60	-	0.40
2	MPA202T	Modern Bio-Analytical Techniques	4	-	-	4	3.0	-	0.60	-	0.40
3	MPA203T	Quality Control and Quality Assurance	4	-	-	4	3.0	-	0.60	-	0.40
4	MPA204T	Herbal and Cosmetic Analysis	4	-	-	4	3.0	-	0.60	-	0.40
5	MPA205P	Pharmaceutical Analysis Practical II	-	12	-	6	-	6.0	-	1.00	-
6	MPA206S	Seminar/Assignment-II	-	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 – II)

L	T	P	C
4	-	-	4

Course Code	**MPA201T**
Course Title	**Advanced Instrumental Analysis**

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 -

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 Learning Outcomes (CLO):

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

1. Understand the fundamental theory of different chromatographic techniques.
2. Describe the principle, instrumentation and applications of HPLC, HPTLC, SFC and GC methods.
3. Discuss the principle, instrumentation and applications of various biochromatographic methods.
4. Explain the instrumentation and applications of various spectroscopic techniques.
5. Predict the structural information using mass and NMR spectrometry and related hyphenated techniques.

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

UNIT I **12 Hours**

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 Hours**

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 Hours**

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 Hours**

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 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 with reference to ^{13}CNMR:

Spin spin and spin lattice relaxation phenomenon. ^{13}C NMR, 1-D and 2-D NMR, NOESY and COSY techniques, Interpretation and Applications of NMR spectroscopy. LC-NMR hyphenations.

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. Kemp, W. *Organic Spectroscopy*. ELBS.
5. Sethi, P. D. *HPTLC: High performance thin-layer chromatography; quantitative analysis of pharmaceutical formulations*. CBS publishers & distributors.
6. Sethi, P. D. *Quantitative Analysis of Drugs in Pharmaceutical Formulations*. CBS Publishers.
7. Munson, J. W. *Pharmaceutical Analysis: Modern Methods (Vol. 11)*. CRC Press.
8. Pavia, D. L., Lampman, G. M., Kriz, G. S., & Vyvyan, J. A.. *Introduction to spectroscopy*. Cengage Learning.

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(M. Pharm - Pharmaceutical Analysis)

(Semester – II)

L	T	P	C
4	-	-	4

Course Code	**MPA202T**
Course Title	**Modern Bio-Analytical Techniques**

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 -

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

Course Learning Outcomes (CLO):

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

1. Understand the basics of drug extraction methods.
2. Determine the biopharmaceutical factors for drug absorption and drug release.
3. Describe pharmacokinetics and its importance along with toxicokinetics.
4. Discuss the principle techniques and applications of various cell culture methods.
5. Apply bioavailability and bioequivalence principles in drug product performance.
6. Predict the possible metabolite formation of drug product.

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

UNIT I **12 Hours**

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 Hours**

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 Hours**

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 Hours**

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.

UNIT V **12 Hours**

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: S

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.

Suggested Readings^: (Latest edition)

1. Chamberlain, J. *The Analysis of Drugs in Biological Fluids*. CRC press.
2. Skoog, D. A., Holler, F. J., & Crouch, S. R. *Principles of instrumental analysis*. Cengage learning.
3. Higuchi, T., Bodin, J. I., & Brochmann-Hanssen, E. *Pharmaceutical analysis*. Interscience Publishers.
4. Munson, J. W. *Pharmaceutical analysis: modern methods (Vol. 11)*. CRC Press.
5. Snyder, L. R., Kirkland, J. J., & Glajch, J. L. *Practical HPLC method development*. John Wiley & Sons.
6. Adamovics, J. A. *Chromatographic analysis of pharmaceuticals (Vol. 74).* CRC Press.
7. Bertholf, R., & Winecker, R. *Chromatographic methods in clinical chemistry and toxicology.* John Wiley & Sons.
8. Weinberg, S. *Good laboratory practice regulations*. CRC Press.
9. Hirsch, A. F. *Good laboratory practice regulations*. Marcel Dekker.
10. ICH, USFDA & CDSCO Guidelines.

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

L	T	P	C
4	-	-	4

Course Code	**MPA203T**
Course Title	**Quality Control and Quality Assurance**

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 -

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

Course Learning Outcomes (CLO):

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

1. Understand the concepts of quality control, quality assurance, GMP, GLP.
2. Describe the various quality control guidelines by CDSCO, USFDA, EMEA, WHO etc.
3. Determine various quality requirements for drugs and finish products.
4. Report various quality related documents for pharmaceutical manufacturing along with quality certification.
5. Relate the importance of operations and controls in pharmaceutical manufacturing.

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

UNIT I **12 Hours**

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 Hours**

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 Hours**

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 Hours**

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.

UNIT V **12 Hours**

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.

Suggested Readings^: (Latest edition)

1. *Quality Assurance Guide by organization of Pharmaceutical Procedures of India*, Volume I & II, Mumbai.
2. Weinberg, S. *Good laboratory practice regulations*. CRC Press.
3. *World Health Organization. Quality assurance of pharmaceuticals: a compendium of guidelines and related materials. Good manufacturing practices and inspection (Vol. 1 & 2)*. World Health Organization.
4. Sharma, P. P. *How to practice GMPs*. Vandana publication Pvt. Ltd Delhi.
5. *World Health Organization. The international pharmacopoeia (Vol. 1 to 5). General Methods of Analysis and Quality specification for Pharmaceutical Substances, Excepients and Dosage forms.* World Health Organization.
6. Hirsch, A. F. *Good laboratory practice regulations*. Marcel Dekker.
7. ICH guidelines
8. ISO 9000 and total quality management
9. Deshpande & Gandhi, N. *The drugs and cosmetics act 1940*. Susmit Publishers.
10. Shah D.H., QA *Manual. Business Horizons*.
11. Willig, S. H., & Stoker, J. R. *Good manufacturing practices for pharmaceuticals. A plan for total quality control. Drugs and the pharmaceutical sciences. Vol. 52*. Marcel Dekker Series.
12. Steinborn, L. *GMP/ISO Quality Audit Manual for Healthcare Manufacturers and Their Suppliers,(Volume 1- With Checklists and Software Package).* Taylor & Francis.
13. Sarker, D. K. *Quality Systems and Controls for Pharmaceuticals*. John Wiley & Sons.

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

L	T	P	C
4	-	-	4

Course Code	**MPA204T**
Course Title	**Herbal and Cosmetic Analysis**

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 -

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 Learning Outcomes:

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

1. Understand the herbal drug regulations and standardization.
2. Identify the adulteration and deterioration of herbal drugs.
3. Analyze the natural products and adulterants.
4. Determine herbal drug-drug interaction.
5. Evaluate cosmetic products.

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

UNIT I **12 Hours**

Herbal remedies- Toxicity and Regulations:

Herbals vs Conventional drugs, Efficacy of herbal medicine products, Validation of Herbal Therapies, Pharmacodynamic and Pharmacokinetic issues.

Herbal drug standardization:

WHO and AYUSH guidelines.

UNIT II **12 Hours**

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.

UNIT III **12 Hours**

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 Hours**

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 Hours**

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.

Suggested Readings^: (Latest edition)

1. Evans, W. C. *Trease and Evans' Pharmacognosy*. Elsevier Health Sciences.
2. Kokate, C. K., Purohit, A. P., & Gokhale, S. B. *Pharmacognosy*. Nirali Prakashan, Pune.
3. World Health Organization. *Quality control methods for medicinal plant materials*. Geneva.
4. Kar, A. *Pharmacognosy and pharmacobiotechnology*. New Age International.
5. Ansari, S. H. *Essential of Pharmacognosy*, Birla publications Pvt. Ltd, New Delhi.
6. Sharma, P. P. *Cosmetics: Formulation, Manufacturing and Quality Control*. Vandana Publications Pvt Ltd. Delhi.
7. *Bureau of Indian Standards. Indian Standard Specification for Raw Materials*. New Delhi.
8. *Bureau of Indian Standards. Indian Standard Specification for 28 Finished Cosmetics*. New Delhi.
9. Harry, R. G. *Harry's cosmeticology*. Chemical Publishing Company.
10. Suppliers catalogue on specialized cosmetic excipients.
11. Butler, H. *Poucher's perfumes, cosmetics and soaps. Springer Science & Business Media.*
12. Barel, A. O., Paye, M., & Maibach, H. I. *Handbook of cosmetic science and technology*. CRC Press.
13. www.who.int
14. www.ayush.gov.in

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

L	T	P	C
-	-	12	6

Course Code	**MPA205P**
Course Title	**Pharmaceutical Analysis Practical II**

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

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 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.

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