### I YEAR I SEMESTER

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<tr>
<th>Subject</th>
<th>Hours/Week</th>
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<td>Bio-Statistics, Intellectual Property Rights &amp; Regulatory Affairs</td>
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<td>Electrometric methods and spectral analysis</td>
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<td>Chromatographic methods of analysis</td>
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<td>Modern Pharmaceutical Analysis Practical – I</td>
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<td>Electrometric and chromatographic methods of analysis-Practical</td>
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<td>Mini-project- I</td>
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### I YEAR II SEMESTER

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<tr>
<td>Advanced Pharmaceutical Analysis</td>
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<tr>
<td>Analytical method development and validation</td>
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<td>Evaluation of dosage forms</td>
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<td>Analytical method development and validation-Practical</td>
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<td>Evaluation of Dosage forms-Practicals</td>
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### II YEAR (III & IV Semesters)

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<tr>
<td>Project work</td>
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MODERN PHARMACEUTICAL ANALYSIS


   b) OPTICAL ROTATORY DISPERSION: Fundamental principles of ORD, cotton effect curves, their characteristics and interpretation. Octant rule and its application with examples. Circular dichroism and its relation to ORD.

3. NMR SPECTROSCOPY: Fundamental principles of NMR (Magnetic properties of nuclei, applied field and precession; absorption and transition; frequency). Chemical shifts concept: Isotopic nuclei, Reference standards: Proton magnetic spectra, their characteristics, presentation terms used in describing spectra and their interpretation (Signal No., Position and Intensity). Brief outline of instrumental arrangements and some practical details. Signal multiplicity phenomenon in high resolution PMR. Spin-spin coupling. Application of Signal split and coupling constant data to interpretation of spectra. De-coupling and shift reagent methods. Brief outline of principles of FT-NMR with reference to 13CNMR. Spin-spin and spin-lattice relaxation phenomenon. Free induction decay (FID) proton noise de-coupling signal, average time domain and frequency domain signals nuclear overhauser enhancement 13CNMR spectra, their presentation; characteristics, interpretation, examples and applications. Brief indication of application of magnetic resonance spectral data of other nuclei by modern NMR instruments. Introduction to 2-D NMR techniques.

4. MASS SPECTROSCOPY: Basic principles and brief outline of instrumentation. Ion formation and types; molecular ion, Meta stable ions, fragmentation processes. Fragmentation patterns and fragmentation characteristics in relation to parent structure and functional groups. Relative abundances of isotopes and their contribution to characteristic peaks. Mass spectrum, its characteristics, presentation and interpretation. Chemical ionization Mass Spectroscopy. GC-MS, other recent advances in MS. Fast atom bombardment mass spectrometry. LC-MS, LC MS-MS.

6. **GAS CHROMATOGRAPHY:** Instrumentation packed and open tubular column, Column efficiency parameters, the Van Deemeter equation, Resolution, liquid stationary phase, derivatization methods of GC including acylation, perfloro acylation, alkylation and esterification. Detectors: FID, ECD, TCD, NPDA. Critical comparison of sensitivity, selectivity and field of applications of these detectors. Examples of GC applications in pharmaceutical analysis.

7. **LIQUID CHROMATOGRAPHY:** Comparison of GC and HPLC, instrumentation in HPLC, analytical, preparative and micro bore columns, normal and reversed phase packing materials, reverse phase HPLC, Column selection, Mobile phase selection, Efficiency parameters, resolution, detectors in HPLC refractive index, photometric and electrochemical. Comparison of sensitivity, selectivity and field of applications of these detectors. HPTLC-instrumentation and applications.

8. **ELECTROPHORESIS:** Moving boundary electrophoresis, Zone electrophoresis, Iontophoresis, PAGE, Isotacophoresis and applications in pharmacy.
   
   **X-ray Diffraction methods:** introduction, generation of X-rays, elementary crystallography, Miller Indices, X-rays diffraction, Bragg’s law, X-ray powder diffraction, X-ray powder diffractometer, obtaining and interpretation of X-ray powder diffraction data. Principle, instrumentation and application of the following: Differential Scanning Colorimetry (DSC), DTA &TGA in analysis of pharmaceuticals.

**REFERENCES:**

3. Instrumental methods of analysis by Willard, Merit, Dean, Settle.
5. Spectrometric identification of organic compounds by Silverstein, Webster.
6. Spectroscopy by B.K.Sharma
7. Fundamentals of analytical chemistry by Skoog
8. Instrumental methods of analysis by Skoog.
10. Organic spectroscopy by William kemp
BIO-STATISTICS, INTELLECTUAL PROPERTY RIGHTS & REGULATORY AFFAIRS

I. BIO-STATISTICS

1. An introduction to statistics and biostatistics—collection and organization of data, graphical, pictorial presentation of data, measures of central tendency and dispersion, sampling techniques, sample size, Coefficient of variation, mean error, relative error, precision and accuracy


3. Design of Experiments: Principles of randomization, replication and local control; CRD, RBD, LSD – their applications and analysis of data; Factorial Experiments – Principles and applications; Probit analysis: Dose – effect relationships, calculation of LD_{50}, ED_{50}.

4. Statistical quality control: Meaning and uses, Construction of $\bar{X}$, R, P, ηp and $\hat{C}$ charts.

II. INTELLECTUAL PROPERTY RIGHTS & REGULATORY AFFAIRS


b). Documentation: Types related to pharmaceutical industry, protocols, harmonizing formulations, development for global filings, ANDA, NDA, CTD, dealing with post – approval changes – SUPAC, handling and maintenance including electronic documentation.

REFERENCES:

5. Applied statistics by S.C.Gupta & V.K.Kapoor
8. Protection of Industrial Property rights, P. Das & Gokul Das
9. Law and Drugs, Law Publications. S.N. Katju
10. Original Laws Published By Govt. of India
11. Laws of drugs in India, Hussain
13. fda.org, wipo.int, patentlawlinks.com, hc-sc.gc.ca, ich.org, cder.org
ELECTROMETRIC METHODS AND SPECTRAL ANALYSIS

Principles, instrumentation and applications of the following spectral and electrometric methods of analysis.

1. UV – visible & IR spectrophotometry
2. NMR Spectrometry
3. ESR Spectrometry
4. Atomic absorption Spectroscopy
5. Plasma emission Spectroscopy
6. Atomic force Microscopy & Photon co-relation spectroscopy
7. Spectrofluorometry
8. Electrometric Methods:
   Conductometry, High frequency titrations, potentiometry,
   Amperometry, Polarography

REFERENCES:

2. Sittle: Handbook of Instrumental Techniques for Analytical Chemistry
JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY ANANTAPUR
M.Pharm Pharmaceutical Analysis & Quality Assurance
I year I semester

CHROMOTOGRAHIC METHODS OF ANALYSIS

Introduction, Principle, Method of preparation and different types of stationary & mobile phases, Instrumentation, Interpretations, Data analysis and its applications of the following

1. Paper Chromatography & Thin layer chromatography
2. Column chromatography & Gas chromatography
3. HPLC
4. HPTLC
5. Exclusion chromatography & Super critical fluid chromatography
6. Vapour phase chromatography & Affinity chromatography
7. Ion – exchange chromatography & Centrifugal partition chromatography
8. LCMS & GCMS

REFERENCES

5. P.D. Sethi ‘Qualitative Analysis of Drugs and Pharmaceuticals
6. I.M. Kolthaf ‘Quantitative Chemical Analysis’
2. UV-Visible spectrum scanning of certain organic compounds- absorption and co-relation of structures, comparisons. Ex: a. Chloramphenicol
   b. Sulphadiazine
   c. Analgin
3. Effect of pH and solvent on UV spectrum of certain drugs.
4. Two dimensional paper chromatography and TLC.
5. Gradient elution and other techniques in column chromatography.
6. Separation by electrophoresis.(PAGE and agarose Gel electrophoresis)
7. Experiments based on HPLC and GC.
8. IR, NMR and Mass spectroscopy of compound each.
9. DSC/XRD curves of a sample and mixture to understand polymorphism.
10. Determination of insulin / any other hormones by ELISA method.
Preparation of Mobile Phase, Standard Solution, Sample Solution, Internal Standard for Acetaminophen, Caffeine and Codeine Phosphate Tablets as per USP.

Test for Identification by using HPLC – For any Compendial Drug

Test for Assay by using HPLC – For any Compendial Drug

Test for Uniformity of Dosage Units by using HPLC – For any Tablets / Capsules as per USP.

Test for Dissolution by using Dissolution Apparatus and HPLC – For any Tablets

Test for Residual Solvents by using GC – For any Compendial Drug

Determination of Alcohol Content by using GC

Test for Assay by using GC - For any Compendial Drug

Test for Identification by using HPTLC – For any Compendial Drug

Test for Assay by using HPTLC – For any Compendial Drug

Test for Impurity Profile by using HPTLC – For any Compendial Drug

Test for Degradation compounds by using TLC – For Acetaminophen as per USP

Part-II

1. Determination of $\lambda_{\text{max}}$ of different drugs and preparation of calibration curve. (Ephedrine Hydrochloride, Sulphanilamide, etc.).

2. Estimation of drugs by UV Spectrophotometry Mimesulide tablets, Paracetamol, Pyridoxine, Pheniramine maleate,

3. Assay of Riboflavin, by Fluorimetry.

3. To study the quenching effect of Quinine sulphate by Fluorimetry.
4. Interpretation of IR spectra of Polystyrene, Salicylic acid, etc.
5. Assay of drugs by IR Spectrophotometry by baseline technique.
6. Interpretation of NMR spectra of pure drugs.
7. Identification of different functional groups by IR
   (amino group, alcoholic group amide, ester, acid group etc)
8. Assay of Cotrimazole tablet by NMR.
   Olive oil, Peanut oil etc.
10. Assay of total zinc in Insulin zinc suspension by Atomic Absorption
    Spectroscopy.
11. Assay of Sodium, Potassium and Calcium in blood serum and water by
    Flame Emission Spectroscopy.
12. Assay of some inorganic agents by Flame Emission Spectroscopy Sodium
    chloride, Potassium citrate, Magnesium acetate.
    Cimetidine, Nitrazepam, Clonidine etc.
15. Study of different fragments of Mass Spectra of different pure drugs.
QUALITY ASSURANCE OF PHARMACEUTICALS

1. a. Concepts of Total Quality Management (TQM) and Good Manufacturing Practices (GMP)


4. Warehousing: Good ware housing practices Materials Management

5. Documentation related to Product Development, Standard operating procedures, standard test procedures, cleaning methods, quality control documents, batch release document, distribution records, complaints and recalls records, retention of records.


8. a. ICH requirements for registration of Pharmaceuticals
   b. WHO certification scheme on the quality of pharmaceutical products.

References

1) Quality Assurance of Pharmaceuticals (A compendium of guidelines and selected materials) Vol I & II (Pharma Book Syndicate, Book Street, Hyd)


3) A guide to Total quality management K. Maitra and S.K. Ghosh

4) Good Manufacturing Practice (GMP) by Mehra

5) How to Practice GMP by P.P. Sharma.


7) Packaging Drugs and Pharmaceuticals W.A. Jenkins & K. R. Osborn.
8) The Drug and Cosmetic Act 1940 by Vijay Malik
9) The International Pharmacopoeia, Vol. 1-4

10) Web links
    a. www.iprlawindia.org
    b. www.inidialegalguide.com
    c. www.intelproplaw.com
    d. www.indianpatentoffice.org
    e. www.findlaw.com
1. Principles and procedures involved in quantitative determination of the following functional groups
   A. Hydroxy  B. Aldehyde  C. Ketone
   D. Amine   E. Methoxy   F. Ester

2. General methods for the estimation of the following
   A. Proteins  B. Carbohydrates  C. Fats
   D. Crude fibre  E. Moisture  F. Nitrogen

3. Principles and procedures involved in the use of the following reagents in Pharmaceutical Analysis
   A. 3- Methyl 1-2- benzothiozoline hydrozone (MBTH)
   B. Folin - Ciocalteau Reagent
   C. Paradimethyl amino benzaldehyde.
   D. 2-6- Dichloro quinine chlorimide
   E. 2,3,5- Triphenyl tetrazolium salt
   F. Ninhydrin Reagent

4. Thermal Methods of Analysis: Theory of Thermal gravimetric Analysis (TGA), Differential thermal Analysis (DTA), Differential Scanning calorimetry (DSC) and Thermal Mechanical Analysis (TMA)

5. Principles and procedures involved in the following physicochemical methods including the assays of official drugs mentioned in IP.
   a) Non-aqueous titration  b) Complexometric titration
   c) Oxidation- reduction titration

6. Principles and procedures involved in the following physicochemical methods including the assays of official drugs mentioned in IP.
   a) Diazotization titration b) Potentiometer titration c) Conduct metric titration

7. a. Radiometric analysis: radio activity, radioisotopes and Pharmaceutical Applications of radiopharmaceuticals
   c. ELISA Test

REFERENCES

1) Remington’s Pharmaceutical Sciences by Alfonso and Gennaro
2) Pharmaceutical Chemistry by Becket and Stanlake
3) Quantitative Analysis of Drugs in Pharmaceutical Formulations by P.D. Sethi
4) Pharmaceutical Analysis by Higuchi, Bechmman and Hassan
5) Theory and Practice of Industrial Pharmacy by Lieberman and Lachman
6) Indian Pharmacopoeia 1996
7) Instrumental Methods of Chemical Analysis by B.K. Sharma
8) A Text Book of Pharmaceutical Analysis by Kerenth A. Conners
9) Journals like Indian Drugs, IJPS etc.
ANALYTICAL METHOD DEVELOPMENT AND VALIDATION

1. Concept of Analytical Method development, validation and calibration of various analytical instruments for drug analysis

2. Development of analytical Methods and validation of the following techniques
   A. UV-Visible spectrophotometer       B. IR Spectrometer       C. Flourimeter

3. Development of analytical Methods and validation of the following techniques
   A. HPLC       B. GC-MS       C. LC-MS

4. A detailed study on related substances and impurities present in drugs and their effect on drug stability and therapeutic action. ICH guidelines for impurity and related substances determination in the drugs

5. Validation Methods for the following
   A. Analytical Procedures       B. Analyst Validation
   C. Air handling equipment and facilities in zone       D. Animal house

6. Validation of sterilization methods and equipment, Dry heat sterilizations, Autocleaving membrane filtration.

7. Validation Methods for the following
   a. Water supplies
   b. Water supply systems: Deionised, distilled, purified, Demineralised and water for injections

8. Accuracy, Precision and Linearity, Sources of Errors, use of significant figures and their correct usage, Intraday and interlay analysis. System suitability and ruggedness of the method

REFERENCES
1) Pharmaceutical Process Validation by Ira R. Berry and Robert A. Nash
2) Quality assurance and TQM for analytical laboratories by M. Parkany, The Royal Society of Chemistry
3) SOP Guidelines by D. H. Shah
4) GMP, by M.L. Mehra
5) A Guide to Total Quality Management by Kaushik Maitra and Sadhan K.Ghosh
6) Microbiological Assays by Barton J. Wright
EVALUATION OF DOSAGE FORMS

1. Detailed study of the principles and procedures involved in various physicochemical methods of analysis of Pharmaceutical dosage forms belong to the following classes of drugs.
   A. Sulphonamides   B. Antibiotics   C. Anti-histamines   D. Vitamins

2. A detailed study on the principles and procedures involved in the determination of the dosage forms of the following group of drugs
   A. Adrenergic   B. Anti-malarial   C. Steroids   D. Analgesics and anti pyretic

3. Official methods of determination for the mentioned below pharmaceutical dosage forms of the following group of drugs
   A. Local anesthetics   B. Barbiturates   C. Anti-diabetic   D. Diuretics

4. Various in process Quality Control tests carried on the following of Dosage Forms
   A. Tablets   B. Capsules   C. Injectables   D. Liquid Orals

5. A detailed study on the biological evaluation of the following dosage forms
   a. Rabbis Vaccine   b. Oxytocin   c. Tetanus Antitoxin

6. Microbiological evaluation of the following dosage forms

7. Quality control of crude drugs: Proximate analysis including ash and extractive values, crude fibre content, UV and Fluorescence analysis of powdered drugs.

8. a. Detection of common adulterants and insects infestation in whole and powdered drugs.
    b. WHO guidelines for the quality control raw materials
    c. Brief study of quality control of plant products and their High throughput Screening

REFERENCES

1) Remington’s Pharmaceutical Sciences by Alfonso and Gennaro
2) Microbiological Assays by Barton J. Wright
3) Pharmaceutical Chemistry by Becket and Stanlake
4) Quantitative Analysis of Drugs in Pharmaceutical Formulations by P.D. Sethi
5) Pharmaceutical Analysis by Higuchi, Bechmman and Hassan
6) Theory and Practice of Industrial Pharmacy by Lieberman and Lachman
7) Indian Pharmacopoeia 1996
Comparison of different methods available for various dosage forms with the official methods mentioned in IP, BP, USP etc.

Method of Determination by colourimetry is compared with the official IP method for its accuracy of the following drugs

1) Paracetamol
2) Aspirin
3) Pipperine
4) Gingiberene
5) Curcuminoids
6) Gingsenosides
7) Sennoides
8) Strychnine and Brucine
9) Caffeine
10) Nicotine
11) Aspiridine
12) Vasicine
EVALUATION OF DOSAGE FORMS - PRACTICAL

1. Assay of Ascorbic Acid Tablets.
3. Assay of trimethoprim and sulphamethoxazole
4. Assay of calcium Gluconate Injection
5. Assay of Penicillin Injection.
6. Assay of Acetyl Salicylic acid tablets.
7. Assay of Ibuprofen Tablets.
10. Assay of Rifampicin Capsules.
11. Assay of INH
Mini Projects:

The mini projects can be taken up as industrial visit/training and report submission. 
Or
A suitable project shall be carried out in the college.

The Project Work:

Separate guidelines will be issued