Maharashtra Education Society's

Abasaheb Garware College

(Autonomous)

(Affiliated to Savitribai Phule Pune University)

Two Year M.Sc. Degree Program in Analytical Chemistry
(Faculty of Science and Technology)

Syllabi under Autonomy
M.Sc. II (Analytical Chemistry)

Choice Based Credit System Syllabus

To be implemented from Academic Year 2023-2024

Title of the Course: M.Sc. II (Analytical Chemistry)

Preamble

The syllabus of Chemistry for Second year has been redesigned for Autonomous Choice based Credit System (CBCS) to be implemented from 2023-2024.

In CBCS pattern semester system has been adopted for M. Sc.-II (Analytical Chemistry) which includes Compulsory and Elective Theory as well as Practical Paper at M.Sc. level. Additional Credit Courses has been introduced at M.Sc. level. Syllabus for Chemistry which includes seven Theory and three Practical subjects for two semester of M. Sc. -II is to be implemented from the year 2023-24. Syllabus for M. Sc.-II will be implemented from the year 2023-24 as per structure approved.

Objectives

1. To impart knowledge of Analytical Chemistry covering all the aspects viz. , Advanced analytical Chemistry, spectroscopy, chromatography, Thermal and electroanalytical methods, Extraction methods, laboratory automation, Analysis of food, polymer and soil.

2. To provide laboratory experience to the students by performing experiments based on topics taught in theory

3. Create awareness and sense of responsibilities towards environment and apply knowledge to solve the issues related to Environmental pollution.

4. Apply knowledge to build up small scale industry for developing endogenous methods

5. Apply various aspects of analytical chemistry to develop interdisciplinary approach

Eligibility : A student should have completed 50% credits of MSc I (Chemistry)

Structure of the Course: Title of the Course

Year	Semester	Course Type	Course Code	Course Title	Remark	Credit	No. of Lectures /Practical to be conducted
2	III	**Theory**	**PSCHA-231**	Electrochemical and Thermogravimetric Methods of chemical analysis		**4**	**60**
		Theory	**PSCHA-232**	Structure Determination and Advanced Extraction Methods		**4**	**60**
		Theory	**PSCHA-233**	Advanced Chromatographic Methods of Chemical Analysis		**4**	**60**
		Theory	**PSCHAELE-234**	A) Analysis of Food and Forensic Chemistry B) Bioanalytical Chemistry		**4**	**60**
		Practical	**PSCHP-235**	Analytical Chemistry Practical -I		**4**	**60**
	IV	**Theory**	**PSCHA-241**	Advanced Analytical Spectroscopic Techniques		**4**	**60**
		Theory	**PSCHA-242**	Chemical Methods of Pharmaceuticals Analysis		**4**	**60**
		Theory	**PSCHAELE-243**	A) Laboratory Automation and Environmental Analytical Chemistry B) Laboratory Automation and Analytical Chemistry of agriculture, polymer and Detergents		**4**	**60**
		Practical	**PSCHAP244**	Analytical Chemistry Practical II		**4**	**60**
		Project	**PSCHAP-245**	Project (Compulsory)		**4**	**60**

SEMESTER-III

PSCHA 211: Electrochemical and Thermogravimetric Methods of Chemical Analysis
(4 Credit- 60 Hours)

Learning Outcomes:

1. Explain instrumentation in electrochemistry and thermogravimetry.
2. describe basic principles of electrochemistry and thermogravimetry.
3. Explain /Describe applications of electrochemistry and thermogravimetry in industry and in analytical laboratory.
4. Apply / select particular method of analysis for sample to be analysed.
5. Solve numerical problems on electrochemistry and thermogravimetry.
6. Interpret polarogram, cyclic voltammogram, pulse polarogram, thermogram, differential thermogram and DSC thermogram.
7. Differentiate among the various methods of electrochemistry and thermogravimetry.

Section I: Electrochemical methods (2 Credit- 30 Hours)

---

1. Coulometry (8 H)

Current voltage relationship during an electrolysis, Operating cell an at fixed applied potential, constant current electrolysis, Electrolysis at constant working electrode potential, Coulometric methods of analysis, Faradays laws of electrolysis, Instrumentations-Constant current and constant voltage instruments, potentiation coulometry-Instrumentation and applications, coulometric titrations - apparatus and applications, problems.

2. Voltammetry and Polarographic Methods of Analysis (16 H)

a) Polarography
b) Pulse Polarography
c) Hydrodynamic Voltammetry
d) Cyclic Voltammetry
e) Amperometry

3. Applications of electroanalytical methods (6H)

Chemically and electrolytically modified electrodes and ultramicroelectrodes in voltammetry, Electrochemical Impedance Spectroscopy

References

1. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.
2. Vogel's Text Book of quantitative analysis 6th Ed.
3. Introduction to Instrumental Analysis by R. D. Braun, Pharmamed Press.
4. Analytical Chemistry, A Modern Approach to Analytical Science, Ed. by R. Kellner, J. M. Mermet, O. Otto, M. Valcarcel, H. M. Widmer, Second Ed. Wiley –VCH

Section-II: Thermal Methods of Analysis Lectures: 30 H

Self learning: Thermogravimetry, Introduction, Instrumentation and Thermogravimetric curves:

1. Differential Thermal Analysis and Differential Scanning Calorimetry (8H)

Introduction, Historical, Definitions: Differential thermal analysis (DTA), Differential scanning calorimetry (DSC), Apparatus: The sensors, The furnace and controller, The computer and display, The reference material, Theory of DTA and DSC, Heat flux DSC, Power-compensated DSC, The effect of higher temperatures, Sample size, Calibration, Applications: Physical changes and measurements, Chemical reactions, Inorganic compounds and complexes, Organic compounds.

2. Thermomechanical and Dynamic Mechanical Analysis (6 H)

Introduction, Definitions: Thermomechanical analysis, Dynamic mechanical analysis, Mechanical moduli, Thermomechanical analysis: Apparatus, Applications: coefficients of expansion, solvent swelling of polymers, phase transitions, sintering), Chemical reactions, Dynamic Mechanical Analysis: Apparatus, Applications: glass transition temperatures, beta and other transitions, relaxation kinetics, polymer miscibility, characterising cross-linking, studying problem samples, characterising film formation.

3. Simultaneous Techniques and Product Analysis (6H)

Introduction, Simultaneous Thermal Analysis: Simultaneous TG-DTA and TG-DSC applications, (sodium tungstate dihydrate, fire-retarded wood, poly(vinyl chloride), pharmaceuticals, reactive atmosphere effects, Evolved gas analysis, Instrumentation: Apparatus, Detection and identification of evolved gases: Physical methods, Chemical methods, Spectroscopic methods (mass spectrometry (MS) and simultaneous TG-MS, calcium oxalate monohydrate, poly (ethylene oxide), brick clays), Infrared and simultaneous TA-infrared, Apparatus, Applications, Gas chromatography and pyrolysis GC-FTIR.

4. Problem Solving and Applications of Thermal Methods (3 H)

5. Radioanalytical Methods of Analysis (7 L)

Neutron activation analysis, principle, technique, steps involved in neutron activation analysis. Radiochemical and instrumental methods of analysis, important applications of NAA. Isotope dilution analysis

References

1. Thermal Methods of analysis, principles, applications and problems, P. J. Haines, Springer-Science Business Media B.V. 1st Ed.
2. Principles of Thermal Analysis and Calorimetry, P. J. Haines, Royal Society of Chemistry
3. Principles and Applications of Thermal Analysis, Paul Gabbott, Blackwell Publishing Ltd. (2008).
4. Thermal Analysis in Practice, Fundamental Aspects, Matthias Wagner, Hanser Publications, 2018.
5. Source book of Atomic energy by Glasstone.
6. Principle of Activation Analysis- P. Kruger, John Wiley and sons, (1971).
7. Nuclear Analytical Chemistry – J. Tolgyessy and S. Verga vol. 2, university Park Press, (1972)
8. Radiochemistry and Nuclear methods – W.D. Ehmann and D.E. Vance, John Wiley and Sons.

PSCHA 232: Analytical Method Development and Extraction Techniques (4 Credit- 60 Hours)

Learning Outcome:

1. Understand various spectroscopic methods for structure determination
2. Explain instrumentations and methodology in spectroscopy
3. Define / understand various terms in analytical extraction and method development and validation.
4. Explain instrumentations and methodology in analytical extraction.
5. Explain / describe basic principles of analytical extraction method development and validation.
6. Explain /Describe applications of analytical extraction and method development in industry and in analytical laboratory
7. Apply / select particular method of analysis for sample to be analysed.
8. Solve numerical problems on analytical extraction

Section-I: Structure determination methods **Lectures : 30 H**

1.IR Spectroscopy: **(6H)**

Principle, Instrumentation, FTIR, Interpretation, Application in structure determination

2. ^{1}H-NMR Spectroscopy: **(8H)**

Instrumentation, Chemical shift, factors influencing chemical shift, deshielding, chemical shift values and correlation for protons bonded to carbons (aliphatic, olefinic, aldehydic, aromatic) and other spin-spincoupling, (n+1) rule, Spin decoupling, Factors affecting coupling constant, simplification of complex spectra, nuclear magnetic double resonance, spin decoupling, contact shift reagents, solvent effects, nuclear over-hauser effect (NOE), resonance of other nuclei like ^{31}P, ^{19}F

3.^{13}C NMR spectroscopy: **(8H)**

FT NMR, Types of 13C NMR Spectra: un-decoupled, Proton decoupled, Off resonance, APT, INEPT, DEPT, chemical shift, calculations of chemical shifts of aliphatic, olefinic, alkyne, aromatic, hetero aromatic and carbonyl carbons, factors affecting chemical shifts, Homo nuclear (^{13}C-^{13}C) and Hetero nuclear (^{13}C-^{1}H)coupling constants.

4.Mass Spectrometry: **(8 H)**

Instrumentation, various methods of ionization (field ionization, field desorption, SIMS, FAB, MALDI, Californium plasma), different detectors (magnetic analyzer, ion cyclotron analyzer, Quadrupoule mass filter, time of flight (TOF). Rules of fragmentation of different functional groups, factors controlling fragmentation, HRMS. Fundamentals, Electron ionization, Chemical ionization, Instrumentation: Quadrupole mass spectrometers, Magnetic sector mass spectrometers, TOF mass analyser, detector; Interpretation of mass spectra, Types of ions Isotopic abundances and characteristic ion clusters, Nitrogen rule and rings-plus-double-bonds, steps in interpretation, Examples

References:

1. Introduction to Spectroscopy – D. L. Pavia, G.M. Lampman, G. S. Kriz, 3rd Ed. (Harcourt college publishers).
2. Spectrometric identification of organic compounds R. M. Silverstein, F. X. Webster, 6th Ed. John Wiley and Sons.
3. Spectroscopic methods in organic chemistry – D. H. Williams and I. Flemming Mc Graw Hill.
4. Absorption spectroscopy of organic molecules – V. M. Parikh
5. Nuclear Magnetic Resonance – Basic Principles- Atta-Ur-Rehman, Springer- Verlag (1986).
6. One and Two dimensional NMR Spectroscopy- - Atta-Ur-Rehman, Elsevier (1989).
7. Organic structure Analysis- Phillip Crews, Rodriguez, Jaspars, Oxford University Press (1998).
8. Organic structural spectroscopy- Joseph B. Lambert, Shurvell, Lightner, Cooks, Prentice-Hall (1998).
9. Organic structures from spectra- Field L. D., Kalman J.R. and Sternhell S. 4th Ed. John Wiley and sons Ltd.
10. NMR spectroscopy of Organic compounds. Jackmann and Sternhell S.

Section II: Advanced Extraction Techniques **Lectures : 30 H**

Self Learning : Classical Approach for Aqueous Extraction Introduction, Liquid-Liquid extraction (LLE), Theory of LLE: distribution ratio and coefficient, solute remaining unextracted, percent extraction, separation factor, factors favouring solvent extraction, quantitative treatment to solvent extraction equilibria, synergistic extraction, extraction reagents for metals, selection of solvents, solvent extraction, problems with LLE process), Examples of Solvent Extraction, Problems.

1.Pre and Post Extraction Consideration (4H)

pre-sampling issues, sampling strategies-solid, aqueous and air, Organic compounds of interest, samples, chromatographic method of analysis, sample preconcentration methods, Liquid-Liquid extraction, synergistic extraction, extraction reagents for metals, selection of solvents, solvent extraction, problems with LLE process), Examples of Solvent Extraction, Reactive extraction, Problems.

2. Solid Phase extraction (SPE) (8H)

Introduction, Types of SPE media, SPE formats and apparatus, method for SPE operation, solvent selection, factors affecting SPE, selected methods of analysis for SPE: applications of SPE, Automation and On-Line SPE and its applications.

3. Solid phase micro-extraction (8H)

Introduction, theoretical considerations, experimental, Methods of analysis: SPME-GC: direct immersion SPME, headspace SPME, analysis of compounds from solid matrix, other SPME-GC application. Methods of analysis: SPME-HPLC-MS. Automation of SPME and its application

4. Solid -Liquid Extraction, Microwave extraction (8H)

New development in micro extraction, Pressurized Fluid Extraction: Introduction, theoretical consideration, Instrumentation for PFE, method development and applications. Microwave assisted extraction: Introduction, instrumentation, Applications

5. Super Critical Fluid Chromatography and Extraction (2H)

Introduction, instrumentation, Applications

References

1. Extraction Techniques in Analytical Science, John R. Dean, Wiley
2. Vogel's Textbook of quantitative Chemical Analysis, sixth Ed., Mendham, Denney, Barnes, Thomas, Pub: Pearson Education.
3. Solid Phase Microextraction, A Practical Guide, Edited by Sue Ann Scheppers Wercinski, CRC press, Taylor and Francis.

PSCHA 213: Advanced Chromatographic Methods of Analysis (4 Credit- 60 Hours)

Learning Outcome:

1.Define / understand various terms in chromatography
2. Explain instrumentations in GC,HPLC,GC-MS and LC-MS.
3. Explain /Describe applications chromatography in industry and in analytical laboratory.
5. Apply / select particular method / instrumental parameters for analysis for sample GC / HPLC.
6. Solve numerical problems on chromatography

Section-I: Gas Chromatography methods **Lectures : 30 H**

---

1. Fundamentals of Chromatographic Methods of Analysis (4H)

2. Gas Chromatography (6H)

Retention data and partition coefficient, separation in the gas phase, Components of gas chromatography: Carrier gas, sample injection, split injection, spitless injection, cold on column injection, programmable temperature vaporization, head space injection, solvent effects, column, detectors- TCD, FID, ECD, Stationary phases for GC: stationary phases for packed column, capillary column, deactivation of surface, different stationary phases, Applications of GC, Problem on quantitative analysis.

3. Gas Chromatography-Mass Spectrometry (10H)

Vacuum and gas flow, Basic principles, Analysis of vacuum and gas flow, Interfaces, Computerization, Computerized operation, Characteristics, Data analysis, Reconstructed gas chromatogram, Mass chromatogram, Selected ion monitoring, Background subtraction, Biller-Biemann stripping technique, Compound identification using reference spectra matching, Mass spectral compilations, Methods of computerized mass spectral search, Commercial mass spectral computer search systems, Quantitative analysis by selected ion monitoring, Choice of ions: basic considerations, Magnetic sector versus quadrupole analysers, Identification and quantitation procedures, Use of isotopically labelled standards, Precision, accuracy and limit of detection, Automated GC-MS operation, Automated data acquisition, Automated data analysis.

4. Applications of GC and GC-MS (4H)

5. Analytical method development and validation (6H)

Method development for chromatographic techniques, Assay Validation and Inter Laboratory Transfer

References

1. Basic Gas Chromatography Mass Spectrometry, Principles and Techniques, F.W. Karasek and R.E. Clement, Elsevier, (Elsevier Science B.V.) 1988
2. Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley –VCH
4. Vogel's, Textbook of Quantitative Chemical Analysis 6th Ed.
5. Standard methods for the examination of water and waste water, 23rd Ed. Rodger Baird, Andrew Eatson, Eugene Rice, jointly published by: Americal Public Health Association, American Water Works Association, Water Environment Federation,
6. Forensic applications of Gas Chromatography by Michelle Carlin and John Dean, CRC press, 2013)
7. Development and validiation of Analytical Methods, Progress Pharmaceutical and Biomedical Analysis, Vol-3, Edited by Chitofer M. Riley and Tomas W. Rosanske (Elsevier).

Section-II: Liquid Chromatography Lectures : 30 H

---

1. Instrumentation of HPLC (8H)

Introduction: HPLC- A powerful separation method, A first HPLC experiment, Liquid chromatographic separation modes, The HPLC instrument, Pumps: General requirements, The short-stroke piston pump, Preparation of Equipment up to Sample Injection: Selection of the mobile phase, Preparation of the mobile phase, Gradient systems, Sample injectors, Sample solution and sample volume; Solvent Properties: Table of organic solvents, Solvent selectivity, Miscibility, Buffers, Shelf life of mobile phases, The mixing cross; Detectors: General, UV detectors, Refractive index detectors, Fluorescence detectors, Electrochemical (amperometric) detectors, Light-scattering detectors, Multiple detection, photodiode array detector; Columns and Stationary Phases: Columns for HPLC, Precolumn, General properties

of stationary phases, Silica, Chemically modified silica, Styrene-divinylbenzene, Column care and regeneration, Introduction to UPLC, comparison between HPLC and UPLC

2. HPLC Methods (10H)

a) Adsorption Chromatography: Normal-Phase Chromatography
b) Reversed-Phase Chromatography
c) Chromatography with Chemically Bonded Phases
d) Ion-Exchange Chromatography
e) Ion-Pair Chromatography
f) Ion Chromatography
g) Size-Exclusion Chromatography
h) Affinity Chromatography
i) Choice of Method

3. Analytical HPLC (2H)

Qualitative analysis, Trace analysis, Quantitative analysis,

4. Separation of Enantiomers (2H)

Introduction, Chiral mobile phases, Chiral liquid stationary phases, Chiral solid stationary phases, Indirect separation of enantiomers. (Ref.-1: 333-345)

5. LCMS Interface and applications (8H)

Interface Technology: Introduction, Thermo-spray interface, The electron spray interface, atmospheric pressure chemical ionization interface and the mechanism of atmospheric pressure chemical ionization. Data acquisition, Processing of mass spectra, Applications of LCMS

References:

1. Analytical Chemistry, Ed. by Kellner, Mermet, otto, Valcarcel, Widmer, Second Ed. Wiley – VCH
2. Practical High-Performance Liquid Chromatography, Veronika R. Meyer, Fifth Ed. John Wiley and Sons, Ltd.
3. Liquid Chromatography Mass Spectrometry: An Introduction by Bob Ardery, Publisher: Wiley India Pvt. Ltd. (2003). A book from series- Analytical techniques in the Science.
4. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.

PSCHA 214: A) Analysis of Food and Controlled Substances (4 Credit- 60 Hours)
B) Bioanalytical Chemistry
PSCHA 214: A) Analytical Methods of Food and Forensic Chemistry

---

Learning Outcome :

1. Define / understand various terms in food analysis techniques and methods, forensic science and drug substances.
2. Explain methods and principles of analysis of Food and drug substances.
3. Select appropriate methods of food analysis for its quality
4. Select appropriate methods for identification of drug and analysis of drug from sample.
5. Solve numerical problems on analysis food and drug substances.
6. Differentiate among the different methods of analysis of food and drug substances.

Section-I: Analytical methods of Food Lectures : 30 H

---

1. Introduction to Food Analysis and sample preparation, Moisture, Total solids and Ash analysis (4H)

2. Analysis of Lipids: (6H)

a) Definition, Classification, General Considerations, Solvent Extraction Methods: Goldfish Method, Semicontinuous Solvent Extraction Method: Soxhlet Method, Discontinuous Solvent Extraction Methods, Nonsolvent Wet Extraction Methods, Babcock Method for Milk Fat (AOAC Method 989.04 and 989.10), Gerber Method for Milk Fat, Instrumental Methods, Comparison of Methods. b) Characterization of Lipids : Estimation of free fatty acids, Saponification value of oils, iodine value, Determination of acid value of oil, determination of peroxide value of oil, p-Anisidine Value and Totox Value, Thiobarbituric Acid Reactive Substances Test, Conjugated Dienes and Trienes, Lipid Oxidation: Evaluating Oxidative Stability, Methods for Lipid Components, Identification and quantification of fatty acids by GC, Problem on quantitative methods.

4. Proteins (6 H)

a. Protein Analysis: Introduction, Classification and General Considerations, Importance of Analysis, Content in Foods, Methods: Following methods with respect to principle, reactions, procedures and applications a) Kjeldahl Method b) Dumas (Nitrogen Combustion) Method, c) Infrared Spectroscopy, d) Biuret Method e) Lowry Method f) Dye-Binding Methods g) Bicinchoninic Acid Method h) Ultraviolet 280nm, Comparison of Methods. b. Protein Characterization Procedures: Amino Acid Analysis, Protein Nutritional Quality: Problem on quantitative methods

5. Carbohydrates analysis by different tests: (6 H)

Extraction, Total Carbohydrate: Phenol-Sulfuric Acid Method, total reducing sugars by Nelson Somyogi method, Specific Analysis of Mono- and Oligosaccharides - High-performance Liquid, Gas Chromatography, Enzymic Methods, Chromatography, Mass Spectrometry, Thin-layer Chromatography, Polysaccharides: Starch, Total Starch,

6. Determination of food preservatives (6 H)

Definition, SO_2 legistration and determination by Tanners method, Nitrate and nitrites legistration and determination, boric acid legistration and determination, Benzoic acid legislation and determination, 4-hydroxybenzoate legislation and determination, ascorbic acid legislation and determination. Sweeteners: Saccharine identification and determination, Colours: Identification by general methods, Natural colours. Problem on quantitative methods.

7. Food adulteration (2H)

References

1. Food Analysis, Edited by S. Suzanne Nielsen, Fourth Edition, Springer
2. Hand Book of Food Analytical Chemistry: Water, Proteins, Enzymes, Lipids, and Carbohydrates by Edited by Ronald E. Wrolstad, Terry E. Acree, Eric A. Decker, Michael H. Penner, David S. Reid, Steven J. Schwartz, Charles F. Shoemaker, Denise Smith, Peter Sporns, Wiley Interscience, a John Wiley & Sons, Inc., Publication.
3. Biochemical Methods, By S Sadashivan, A. Manickam; Third Edition, New Age International Publishers
4. Pearson's Chemical Analysis of Food

Section-II: Analytical Methods of Controlled Substances Lectures : 30 H

---

1. Forensic language (3H)

Definition: Drug, Drug abuse, Controlled Substance Statutes

2. Forensic documentation (3H)

3. Chemical Screening and Microcrystal Tests (6H)

a) Chemical tests: Introduction, Chemistry of Color Formation, Limitations of Chemical Color Tests, Chemical Color-Test Methods, Documentation, Chemical Colour Tests:

Chen's Test, Dille–Koppanyi's Test, Mecke's Test , Marquis' Test, Nitric Acid Test, Primary Amine Test, Secondary Amine Test, Tertiary Amine Test, Van-Urk's Test, Duquenois–Levine Test, Froehde's Test, Janovsky Test, Weber Test. b) Microcrystal Techniques: Introduction, Advantages of Microcrystal Techniques, Disadvantages of Microcrystal Techniques, Documentation, Microcrystal Test Techniques, Aqueous Test Technique, Volatility Test Technique, Acid and Anionic Test Technique, Aqueous Test Reagents,

3. Analysis of Drugs/Narcotics (12 H)

a) Amphetamine and Related Compounds b) The Analysis of LSD c) Cannabis sativa and Products d) Diamorphine and Heroin e) Cocaine f) Products from Catha edulis and Lophophora williamsii g) Analysis Barbiturates and Benzodiazepines

4. DNA profiling and Fingerprint analysis (6H)

DNA and its polymorphism, DNA typing procedures-RFLP, PCR, MVR-PCR, Dot-blot, AMP-FLP, STR, other methods, paternity testing, applications, interpretation and practical use, Latent fingerprints; optical, physical, physico-chemical & chemical detection methods; fingerprints in blood, fingerprint detection sequences.

Reference

1. Textbook of Forensic Pharmacy, C. K. Kokate, S. B. Ghokhale, Pharma Med Press (2008)
2. Textbook of Forensic Pharmacy, B. M. Miital
3. Basic Principles of Forensic Chemistry, Javed I. Khan • Thomas J. Kennedy Donnell R. Christian, Jr. Humana Press
4. Analysis of Controlled Substances, Michael D. Cole, Wiley (2003)
5. Forensic Chemistry' by Suzanne Bell, Pearson Prentice Hall Publishers, 2006

PSCHA 234: B) Bioanalytical Chemistry (4 credit, 60 H)

Learning Outcome:

1. Define / understand various terms in Electrophoresis, capillary electrophoresis, HPTLC, Body fluid analysis, ELISA, RIA.
2. Explain instrumentations in in Electrophoresis, capillary electrophoresis, HPTLC, Body fluid analysis, ELISA, RIA.

Section-I: Bioanalytical Techniques (Lectures, 30 H)

---

1. Introduction to Electrophoresis (8H)

General introduction to Electrophoresis: Introduction and applications of electrophoresis; Types of electrophoretic systems: Moving boundary electrophoresis, Zone electrophoresis, Steady state electrophoresis; Support media in Zone electrophoresis: filter paper, cellulose acetate, gel media; Factors Affecting Electrophoretic Mobility: Characteristic of charged molecules, Characteristic of the electrophoretic system; Detection in electrophoresis: optical methods, radiochemical methods, biological assay methods

2. Capillary Electrophoresis: Basics, Instrumentation and Application (12H)

a) Basic Principles: Basic Electrophoretic Separation Modes, Zone Electrophoresis, lsotachophoresis, lsoelectric Focusing, Set-up for Capillary Electrophoresis, Theory of Electrophoretic Migration, Determination of Effective Mobility, Electroosmosis, Performance Criteria, Efficiency, Resolution.

b) Instrumentation: Injection, Hydrodynamic Injection, Electro-kinetic Injection, General Aspects of Injection, Detection, General Aspects, Evaluation of Detector Performance, UV -VIS Absorbance Detection, Light Sources for UV -VIS Detection, Optical Layout of a UV -VIS Detector for CE, Design of the Detection Cell, Fluorescence Detection: Excitation Sources for Fluorescence Detection, Optical Layout of a Fluorescence Detector, Derivatization with

Fluorescent Tags, Pre- and Post-Column Derivatization, Electrochemical Detection, Conductometric Detection, Amperometric Detection, Capillary Column, Sample Collection, Commercial Instruments.
c) Qualitative and Quantitative Analysis and Applications: General Aspects of Qualitative and Quantitative Analysis, Application: Drugs and Natural Products, Amino Acids, Peptides and Protein.

3. HPTLC and Detectors for HPTLC (10H)

Thin layer chromatography, High performance thin layer chromatography.
Planar Chromatography Detectors, Transmittance Measurements in Thin-Layer Chromatography, The Lambert-Beer Law, Reflectance Measurements in TLC and HPTLC, The Kubelka–Munk Equation, Reflectance Measurements with a Diode-Array Scanner, Spatial Resolution on the Plate, Spectral Distribution on HPTLC Plates, Spectral Evaluation Algorithm, Mass Spectrometric Detection in TLC, Direct Plate Extraction (SSSP), MALDI Techniques (MALDI-MS), Atmospheric Pressure Mass Spectrometry. Applications.

References:

1. Electrophoresis, Analytical chemistry through open learning Series, Wiley
2. Capillary Electrophoresis: Principles and Practice, R. Kuhn S. Hoffstetter-Kuhn, Springer Laboratory, Springer-Verlag
3. Vogels's Textbook of Quantitative Chemical Analysis, 6th Ed.
4. Quantitative Thin-Layer Chromatography-A Practical Survey, Bernd Spangenberg, Colin F. Poole, Christel Weins, Published by Springer

---

Sec-II: Clinical Analytical Chemistry Lectures : 30 H

1.Analysis of blood and urine (14H)

a) Collection of Specimens: Blood: Collection of Blood specimens, storage and preservation, Urine: Collection of Urine, physical characteristics of urea, preservation and storage, Faeces: Collection and preservation. b) Analysis of Blood and urine: Determination of blood and plasma glucose by glucose oxidase method, Determination of urine for glucose, Determination of ketone bodies in blood, Oral Glucose tolerance test, Determination of serum creatinine, estimation of serum bilirubin, Estimation of serum cholesterol, determination of blood haemoglobin, Urate: determination of serum urate, Determination of urea in urine by urease method and by direct colorimetry, Estimation of Na, K, Ca by flame photometry, inorganic phosphate by colorimetry. c) Determination of vitamins in body fluid: Classification of vitamins with example, Each vitamin must be explained with respect of functions, deficiency diseases, daily requirement, and analytical method i) Retinol (determination of retinol and serum carotene in serum using TFA), Vit D3 (cholecalciferol), Vitamin E (Tocopherols, Determination of serum tocopherol by spectrophotometry by dipyridyl method), Vitamin B1 (thiamine determination by flurometry), Vitamin B2 (riboflavin, Photofluorometric method), Vitamin B6 (Pyidoxine, Fluorometric determination of Xanthuric acid), Nicotinic acid and Niacin: determination by fluorometry, Ascorbic acid (vitamin –c) Volumetric method using 2,6 dichlorophenol method, colorimetric determination of leucocyte ascorbate. (Ref.-1, Relevant pages)

2. Immunological methods of analysis (12 H)

a) Basic of immunology: The immune response, Antigen, Adaptive Immunity and Clonal Selection, Antibodies, Antigen (Antibody production in response to antigen stimulus, affinity and avidity, antibody production in response to immunization vaccination, Antibody production in response to infectious agents, relation between antigen and antibody in vivo, diagnostic usefulness of antigen and antibody in infection disease), Antigenic Commonness,

b) Basic Principles of ELISA: Reactions scheme, Direct ELISA, Indirect ELISA, Sandwich ELISA, Competition EILISA, Choise of Assay, Stages in ELISA: Solid phase (Immobilization of antigen on solid phase coating, coating time and temeperature, coating buffer, desorption, binding capacity, nonspecific binding, covalent antigen attachment), Washing, Addition of reagents, incubation, blocking conditions and non-specific reactions, enzyme conjugates, conjugation with enzymes, Development of label, stopping reactions, reading. Practical Exercise for Direct ELISA: Explain with respect to learning principles, reaction scheme, basis of assay, materials and equipment's, practical details, data explained, aspects of assay described, conclusions. The pregnancy test on urine. (Ref-2, 3)

3. Radioimmunoassay (4H)

Radioimmunoassay (RIA), Principle, RIA Reagents, RIA Steps, RIA Results Interpretation (Ref-1, 4)

References: 1. Varley's Practical Clinical Biochemistry, Gowenlock A. H., 6th Edition, 2006, CBS Publishers, New Delhi. 2. Methods in Molecular Biology, Vol-42, ELISA-Theory and Practice, by John R. Crowther, Humana Press, Totowa, New Jersey.
3. Enzyme-linked Immunosorbent Assay (ELISA) From A to Z, Samira Hosseini, Patricia Vázquez-Villegas, Marco Rito-Palomares, Sergio O. Martinez-Chapa, published by Springer,
4. Basic Serological Testing, Rowa Yousef Alhabbab, Published by Springer

---

PSCHA 235: Basics of Instrumental Methods of Chemical Analysis (4 Credit- Hours)

Learning Outcome:

1. Maintain proper record of analytical data in notebook. Observe personal safety in laboratory and able handle all chemicals, instruments, etc safely in laboratory.
2. Define / understand various terms involved practical methods of quantitative analysis.
3. Explain instrumentations of colorimeter, spectrophotometer, photoflurometer, TGA, HPLC, GC, Flame-photometer, CV, AAS, etc.
4. Explain / describe basic principles of chromatography different instrumental methods of analysis.
5. Design / modify and validate new analytical method for chemical analysis of particular sample.
6. Apply / select particular method / instrumental parameters for analysis of given sample.
7. Give mathematical treatment to analytical data and able to interpret the results accurately.

Section-I: Analytical method Development and Validation (Any 12 experiments)

---

1. Demonstration Practical: a. Calibration of UV-Visible spectrophotometer for control of absorbance as per IP or BP b) Theoretical basis for the choice of solvent for recording UV-Visible spectra of substances c) Theoretical basis for choice proper concentration for recording the UV-Visible spectrum d) Recording the UV-Visible spectrum of any one substance like caffeine, aspirin, paracetamol, $KMnO_4$ or any other substance of interest having characteristic UV-Visible absorbance and i) identification of characteristics peaks in spectrum, b) Choice of λmax for quantitative analysis c) Calculation of Molar absorptivity (ε) and d) Sp. absorbance

2.Table Work: a) Theorical basis of method development and validation – Accuracy, precision, noise level, detection limit, quantitation limit, Calibration curve and standard addition method and theoretical basis of choice between two, b) Expression of results:

Calculation of mean, standard deviation, error and absolute error, elimination of data, c) Regression analysis of calibration curve and its importance.

3-6. Analytical method development and validation

Study of visible spectroscopic or colorimetric method for estimation of particular metal ion or non-metal ion or organic substance with respect to: a) Selection of ligand / reagent and colour formation method b) Choice of reaction cond. such as concentration of analyte and colour forming reagent, pH for colour formation reaction, etc. c) Determination of λmax for quantitative analysis d) estimation of noise level, detection limit, quantisation limit and linearity range (Calculate R^2 value). Thereby set conc. limits for calibration curve method and standard addition method. e) Estimation of known of metal ion by calibration curve method and by standard addition method in triplicate for the validation of method. f) Estimation of metal ion from sample by calibration curve method and by standard addition method in triplicate (Regression analysis must be performed for both methods and results shall be accepted when R^2 is greater than 0.95) g) Detection of possible interfering metal ion.

7. Analysis of Riboflavin by visible spectrometry and Photoflurometry. Compare results with respect to sample requirement, detection limit, accuracy of both methods. Give your choice for analysis of i) Riboflavin as raw material in pharmaceutical industry andii) blood/ urine/vitamin supplement. Explain reason for choice of method. (Ref-4, 6 and 9).

8. Comparison of end point redox titration between $K_2Cr_2O_7$ and standard Fe(II) i) by potentiometry and ii) external indicator. Calculate amount of Fe(II) by both methods and compare with standard value. Give critical comment on Fe(II) content by two methods with respect to standard value i.e. accuracy of results and advantages and disadvantages of each method.

9. Assay of local anaesthetic (benzocaine) by non aqueous titration method

10.Determination of glucose from glucon D by titration with Fehling solution

11. Determine aspirin in tablet conventional titration and instrumental method and compare the results of two method.

12. Analysis of waste water /natural water sample for pH, dissolved oxygen, total dissolved salts, Turbidity, colour, Total hardness Alkalinity and Buffering capacity of water

13. Determination of Cr(VI) by diphenyl carbazide method.

14. Determination of total casein and lactose in milk

15. Adulteration Test for Milk and Milk product

16. Determination of alcohol from given sample by Spectrophotometrically

References:

1. Separation, Preconcentration and Spectrophotometry in Inorganic Analysis, by Z. Marczenko and M. Balcerzak, Analytical Spectroscopy Library – 10, Elsevier

2. Standard methods for the examination of water and wastewater, 23rd Ed. Roger B. Baird, Andrew D Eaton, Eugene W. Rice, American Public Health Association, Americal water works association, Water environment federation.

3. Vogels textbook of Inorganic Quantitative Analysis,

4. Biochemical Methods, Third Edition, By S Sadashivan, A. Manickam; New Age International Publishers

6. Indian Pharmacopeia: 2007, Vol-1, 2, 3.

7. Chemical Analysis and Material Characterization by spectrophotometry, Bhim Prasad Kafle, Elsevier

8. Ultraviolet and Visible Spectrophotometry in Pharmaceutical Analysis, Sandor Gorog, Published by CRC press, Taylor and Fransis.

9. An introduction to Practical Bichemistry, David T. Plummer, Tata McGraw-Hill publishing Company Ltd.

10. Experiments in chemistry, D. V. Jahagirdar, Himalaya Publishing Company
11. Method Development for Analysis of Aspirin Tablets, Journal of Chemical Education, Volume 65 Number 10 October 1988.
12) Vitamin C as a Model for a Novel and Approachable Experimental Framework for Investigating Spectrophotometry, Journal of Chemical Education, DOI:10.1021/acs.jchemed.9b00197

Section – II: (Any12 experiments)

---

1. Estimation organic nitrogen by Kjeldahl's Method or semi-micro Kjedahl's method
2. Extraction of organic substance by Soxhelet or semi micro Soxhelet extraction
3. Isolation of carotenoids from spinach / lycopene from tomato. TLC / column separation to find out number of carotenoids. (Ref-6)
4. Determination of Ion exchange capacity of ion exchange resins (Ref-1)
5. Solid Phase Extraction: Isolation of amino acids from aqueous sample using ion exchange resin and their identification by colorimetric test (Ref. 5)
   Or Isolation of organic compound using SPE cartridge and quantitative estimation (Ref-7)
6. Pre conc. using solid phase extraction on ion exchange cartridge and estimation.
7. Flame photometric analysis of water /soil sample for Na+ and K+ by calibration curve method (give regression analysis for both curves) (Ref-1)
8. Estimation of K+ from soil/water sample by internal standard and its confirmation by standard addition method (give regression analysis of both curves) (Ref-1)
9. Selective estimation of Cl^- from water or saline sample or food sample by calibration curve method using turbidimetry (give regression analysis) and its confirmation by standard addition method. (Ref-1)
10. Selective estimation of SO_4^{2-} in presence of chloride from water sample or any other sample by calibration curve and its confirmation by turbidimetric titration method (give regression analysis for both curves).

11.Estimation of quinine sulphate from tablet by calibration curve and its confirmation by standard addition method.

12. Determination of glucose in DNS saline and glucose supplement (Glucon-D) sample by polarimeter.

13. Estimation of Vit. C using Dichlorophenol, Indophenols by volumetric method

14. Separation of Colours by TLC / Paper chromatography, their isolation by elution from paper or TLC and quantification by colorimetry.

15. Estimation of Iron from syrup

16.Demonstration of HPLC by Mentor

17. Demonstration of GC by Mentor

18. Demonstration of TGA by Mentor

19.Demonstration of AAS Practical by Mentor:

Students Self activity

1.Construction of any one working electrode and perform redo titration using electrode prepared.

2. Applied Qualitative Analysis - Toxicological Test (Ref-10)

Qualitative and confirmatory test

Test for aniline / para aminophenol, Test for antimony / mercury (No C.T.), Test for Borate (use talcum powder), Dinitrophenol pesticides, Ethanol / methanol, Formaldehyde, Hypochlorites, Iodates, Nitrate / nitrite, Paracetamol, Phenol, Salicylic acid its derivatives, Thiocynates

3.Internship in any laboratory related to analytical chemistry

References:

1. Vogel's Textbook of Quantitative Chemical Analysis, 6th Ed.
2. Indian Pharmacopeia, 2007
3. Chemical Separations Principle techniques and Experiments, Clifton E Meloan, Wiley Interscience.
4. Separation, Preconcentration and Spectrophotometry in Inorganic Analysis, by Z. Marczenko and M. Balcerzak, Analytical Spectroscopy Library – 10, Elsevier
5. Standard methods for the examination of water and wastewater, 23rd Ed. Roger B. Baird, Andrew D Eaton, Eugene W. Rice, American Public Health Association, Americal water works association, Water environment federation.
6. Biochemical Methods, Third Edition, By S Sadashivan, A. Manickam; New Age International Publishers
7. Extraction technique in Analytical Science, John R. Dean, Wiley
8. Experiments in modern analytical chemistry, D. Kealey, Springer Science Business media, 1986.
9. Student Construction of a Gel-Filled Ag/AgCl Reference Electrode for Use in a Potentiometric Titration, Journal of Chemical Education, Vol. 76 No. 1 January 1999
10. Basic Analytical Toxicology, R. J. Flangan, R. A. Braithwait, S. S. Brrown, B. Viddop, F. A. de Wolff, published by WHO

Semester-IV

PSCHA 241: Advanced Analytical Spectroscopic Techniques (4 Credit- 60 Hours)

Learning Outcome

1. Define / understand various terms and principles in atomic absorption, atomic emission, fluorescence,
ESR and electron spectroscopy.
2. Explain instrumentation of atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.
3. Explain advantages of ICPAES-MS over AES spectroscopy, fluorescence spectroscopy.
4. Solve numerical problems on analysis all these spectroscopic methods.
5. Interpret ESR spectra, super hyperfine splitting and g value in ESR, and parameters affecting it.
6. Calculate theoretical parameters from ESR data and characterize compound.
7. Solve problems based on atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.

Section-I: Atomic Spectroscopic Methods (Lecture 30H)

Self Learning :Sample preparation techniques

Introduction, aqueous sample, liquid-liquid extraction, Ion exchange, co-precipitation, solid samples: decomposition techniques, microwave digestion, dry ashing, fusion, Extraction procedures: Single extraction, sequential extraction, enzymatic digestion

1. Applications of Atomic Absorption and emission Spectroscopy (6H)

Recapitulation Theory, Instrumentation of AAS and Flame photometry

Applications of AAS and FES in environmental, pharmaceutical and material analysis, Analysis of Na and K flame photometry, determination of Ca and Mg in tap water, determination of Pb in ferrous alloy, Determination of trace elements in contaminated soil, Analysis of metals from waste water sample of AAS method

2. Inductively Coupled Plasma AES and MS (12 H)

Inductively Coupled Plasma AES: Introduction, inductively coupled plasma, Direct current plasma, microwave induced plasma, glow discharge, plasma spectroscopy, spectrometers, Detectors, interferences. Inductively Coupled Plasma MS: Fundamental of MS, Inorganic mass spectroscopy, Interface, mass spectrometer, quadrupole mass analyser, detectors, interferences, isotope dilution analysis, mass spectral interpretation. Applications: Forensic analysis of documents, analysis of coal, Clinical analysis of blood and urine, environmental analysis-soil, food analysis – milk products. Analysis of metals from waste water sample of ICP-MS method.

3. Atomic Fluorescence Spectroscopy (8H)

Atomic fluorescence, Apparatus for AFS, EMR source for AFS, LASERS, Cells for AFS, Plasmas-ICP and DCP, Detectors, theory of AFS, Analysis with AFS, Interferences with AFS, Resonant ionization Spectroscopy, LASER enhanced ionization spectroscopy.

4. Elemental Analysis (4H)

Particular analyses, elemental organic microanalysis, total nitrogen analysers (TN) total sulpur analysers, total carbon analysers, Problems on empirical and molecular formula on CHONS analysis.

Reference

1. Practical Inductively Coupled Plasma spectroscopy, John R. Dean, Wiley India Pvt. Ltd. (AnTs Series book)
2. Standard methods for the examination of water and waste water, 23rd Ed. Jointly published by American Public Health Association, American Water Work Association, Water Environment Federation. 2017.
3. Vogels, Quantitative Chemical Analysis, 6th Ed.
4. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.
5. Introduction to Instrumental Analysis by R. D. Braun
6. Practical Guide to ICP-MS, Edited by Robert Thomas, CRC press, Francis and Taylor.

Section-II: Molecular Spectroscopic Methods (Lectures 30 H)

---

1. Molecular Luminescence spectrometry (8H)

Introduction, theory of fluorescence and phosphorescence: excited state producing fluorescence and phosphorescence, energy level diagram, rate of absorption and emission, deactivation process, variables affecting fluorescence and phosphorescence, Emission and excitation spectra, Instruments for measuring fluorescence and phosphorescence, Instrument standardization and applications. Chemiluminescence: The Chemiluminescence phenomenon, measurement of chemiluminescence, analytical applications, problems.

2. Electron Paramagnetic Resonance Spectroscopy (14 H)

Basic Theory: general remarks, electron spin and magnetic moment, ESR transitions, Selection rules, g-factor, presentation of spectra, interaction of magnetic dipole with microwave radiations, Larmor precision, resonance phenomenon, relaxation process, transition probability. Hyperfine Structure: Nuclear hyperfine splitting, radical containing one proton, spin Hamiltonian, selection rules, radical containing a set of equivalent protons, radical containing a set of multiple protons, radical containing multiple sets of protons ($I = ½$), radical containing multiple sets of proton ($I > ½$), Atomic radicals, Origin of hyperfine interaction, sigma radicals, assignments of spectra using Huckel MOs, alternant hydrocarbons, hyperfine splitting constants, second order splitting, Applications.

3. Electron Spectroscopy for Surface Analysis (8H)

Basic principles, x-ray photoelectron spectroscopy, Auger Electron spectroscopy, Instrumentation: ultra-high vacuum, source gun, electron gun, Ion gun, electron energy

analysers, Characteristics of Electron spectra: photoelectron spectra, Auger electron spectra, Qualitative and quantitative analysis: qualitative analysis, peak identification, chemical shift, problems with insulating materials, Quantitative analysis: peak and sensitivity factor, composition depth profiling.

References:

1. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.
2. NMR Spectroscopy Explained, Neil E. Jacobsen, Wiley Interscience (A John Wiley and Sons Inc., publication)
3. Materials Characterization, introduction to microscopic and spectroscopic techniques, Yang Leng, 2nd Wiley-VCH.
4. Introduction to Magnetic Resonance of Spectroscopy ESR, NMR, NQR, D.N. Sathyanarayana, I. K. International Publishing House Pvt. Ltd.

PSCHA 242: Chemical Methods of Pharmaceuticals Analysis (4 Credit- 60 Hours)

Learning Outcome

1. Define / understand various terms in pharmaceutical raw material and finished product analysis.
2. Explain various pharmaceutical dosage forms and types of raw materials used.
3. To describe basic principles of methods of pharmaceutical analysis according to IP.
4. Understand ICH guidelines and role of FDA.
4. Explain importance particular test in pharmaceutical raw material and finished product analysis.
5. Perform and explain importance of limit tests, identification tests and test of raw materials and finished products.
6. Solve numerical problems on analysis pharmaceutical raw material and finished product analysis.
7. Interpret IR spectra, HPLC chromatogram, UV-Visible spectra of pharmaceutical materials.

Section-I: Pharmaceutical Dosage forms and General Methods Analysis (Lectures 30 H)

---

1. FDA and ICH Guidelines (3H)

Role of FDA, Introduction to New Drug, Development of New Drugs- Selection of Area,, Phase I, Phase II, Phase III, Application to FDA for formulation and marketing for new drug,ICH guidelines

2. Good Manufacturing process and Good Laboratory Process (3H)

3. Pharmaceutical Dosage Forms (8H)

Capsules: Definition, types of capsules, Tests; Creams: Definition, tests; Ear Drops: Definition, tests; Eye Drops: Definition, tests; Gels: Definition, Inhalation Preparations: Definition, Uniformity of delivered, Number of deliveries per container dose, Uniformity of delivered dose (only); Nasal preparations: Definition and tests; Ointments: Definition and tests; Oral Liquids: Definition, types and tests; Oral Powders: Definition and tests; Parenteral Preparations: Introduction, Injections: Definition and tests, Infusion: Definition and tests;Powder for Injection: Definition and tests; Tablets: Definition, types of tablets and their tests, Shelf life of pharmaceutical preparation.

4. Chemical Test, Limit Test and Assay (8 H)

Important Note: Write the chemical reaction and explain theoretical basis of the limit tests and assay though it is not given in reference book.

a) Limit Tests: Aluminium, Aluminium in Adsorbed Vaccines, Arsenic, Calcium in Adsorbed Vaccines, Chlorides, Heavy metals, Iron, Lead, Potassium, Sulphates, Sulphated Ash, Total Ash, Free Formaldehyde, N-N-Dimethylaniline

b) Assays: Acetyl Value, Acid Value, Cineole, Ester, Ester Value, Hydroxyl Value, Iodine value, Nitrogen, Methoxyl, Nitrite Titration, Peroxide Value, Saponification Value, Assay of Steroids, Unsaponifiable Matter, Assay of Vitamin A, Assay of Vitamin D, Water, Zinc, Ethanol, Assay of Insulins

5. Pharmaceutical Methods of Determination (8H)

Disintegration Test, Dissolution Test, Uniformity of Weight of Single-Dose Preparations, Uniformity of Content of Single-Dose Preparations, Friability of Uncoated Tablets, Contents of Packaged Dosage Forms, Powder Fineness, Particle Size by Microscopy, Particulate Contamination. Stability Testing, Shelflife expiry date

Section-II: Analysis of Raw Materials and Active Ingredients (2 credit, 30 H)

1. Introduction to Pharmaceutical Analytical Chemistry (2H)

Introduction, Official European Pharmacopoeia definitions, Pharmaceutical Analytical Chemistry, Manufacture of Pharmaceuticals, Development of New Drugs,

2. Chemical Analysis of Pharmaceutical Ingredients (12 H)

Pharmaceutical Ingredients, Production, and Control, Pharmacopoeia Monographs, Melting point capillary method, Impurities in Pharmaceutical Ingredients,Identification of Pharmaceutical Ingredients: IR Spectrophotometry, UV-Vis Spectrophotometry, Thin-Layer Chromatography, Melting Point, Optical Rotation, Liquid Chromatography, Chloride and Sulfate, Identification, Impurity Testing of Pharmaceutical Ingredients, Related Substances Residual Solvents, Foreign Anions, Elemental Impurities, Loss on Drying, Water, Identification and Impurity Testing of Organic Multi-Chemical Ingredients: Oxidizing Substances, Assay of Pharmaceutical Ingredients, Aqueous Acid–Base Titration, Non-Aqueous Acid–Base Titration, Redox Titrations, Liquid Chromatography, UV-Vis Spectrophotometry,

3. Chemical Analysis of Pharmaceutical Preparations (12H)

Chemical Analysis of Pharmaceutical Preparations, Monographs and Chemical Analysis (BP Identification of the API: Identification by IR Spectrophotometry ,Identification by Liquid Chromatography, Identification by UV-Vis Spectrophotometry, Assay of the Active Pharmaceutical Ingredient, Assays Based on Liquid Chromatography, Assays Based on UV Spectrophotometry, Assays Based on Titration ,Chemical Tests for Pharmaceutical Preparations, Test for Related substances, Uniformity of Content, Dissolution.

4.Impurity Profiling (4H)

Designations of Impurities, Regulatory Requirements, Isolation Methods, Characterization Methods, Case Studies, Role of Drug Master Files (DMF)—Type II and Impurities Evaluation, Reference Standards for the Quantitation of Impurities and Analytical Procedures, Qualification of Impurities and New Impurities

References

1. Indian Pharmacopeia Volume I, 7th Ed

2.Indian Pharmacopeia Volume II, 7th Ed

3. Introduction to Pharmaceutical Analytical Chemistry, Stig Pedersen-Bjergaard, Bente Gammelgaard, Trine Grønhaug Halvorsen, Second Edition, Wiley (2012).

4. ICH Quality Guidelines: An Implementation Guide, Andrew Teasdale, David Elder, Raymond W. Nims, Wiley, Year: 2017

5.HANDBOOK OF ISOLATION AND CHARACTERIZATION OF IMPURITIES IN PHARMACEUTICALS Satinder Ahuja Ahuja Consulting Calabash, NC Karen Mills Alsante,Elsevier, 2003

6. Euroean pharmacopoeia

7. Practical Pharmaceutical chemistry third edition volume 1. By A.H.Beckett &J.B.Stenlake.
8.Remington's Pharmaceutical sciences.

PSCHA 243: A) Laboratory Automation and Analytical Chemistry of agriculture, Polymer and Detergents (4 Credit- 60 Hours)
Or
PSCHA 243:B) Laboratory Automation and Environmental Analytical Chemistry

---

PSCHA 243: A) Laboratory Automation and Analytical Chemistry of agriculture, Polymer and Detergents

---

Learning Outcome

1. Define / understand various terms in Laboratory automation and sensors
2. Explain instrumentation of automated laboratory analysis and sensors.
3. To describe basic principles of automated laboratory analysis and sensors.
4. Explain importance of automated laboratory analysis and sensors.
5. Define / understand various terms in soil analysis, pesticide residue analysis, detergent analysis and polymer analysis.
7. To describe basic principles techniques / methods of soil analysis, pesticide residue analysis, detergent analysis and polymer analysis.
8. Explain importance of soil analysis, pesticide residue analysis, detergent analysis and polymer analysis.
9. Choose suitable method / techniques to characterize quality of soli polymer and detergent.
10. Describe / explain results of analysis soil, pesticide residue, detergent and polymer.
11. Solve numerical problems on analysis soil, pesticide residue, detergent and polymer.

Section-I: Laboratory Automation and Sensor Based Techniques (Lecture: 30H)

---

1. Introduction to laboratory Automation (2H)

Introduction, automation, miniaturization and simplification, lab automation, flow injection analysis, miniaturized analytical systems, fast response analytical systems, chemical sensors, screening systems, process on-line systems.

2. Laboratory Automation (6H)

Definition and concept, objective of automation in analytical chemistry, automation of analytical tools and process, automation of preliminary operations, automation of calibration, automation of measuring and transducing of analytical signals, automation of data acquisition and processing, analysers, automated management system, advantages and shortcomings of automated system.

3. Flow Injection Analysis (6H)

Batch and continuous flow analysis, principles, basic FIA instrumentation, dispersion in FIA, FIA for reproducible and precise sample preparation, FIA system with enzymes, flow injection hydride generation scheme, online sample conditioning, and preconcentration, exploiting the physical dispersion process, FIA gradient technique, Process control, process control analysers.

4. Miniaturized Analytical systems (6H)

Introduction, Concept, theory of miniaturization, microfabrication, silicon and glass micro-matching, polymer replication technology, miniaturized analytical components, sampling and sample pre-treatment, system integration, serial integration, parallel integration, commercialization.

5. Chemical Sensors (6H)

Introduction, definitions, Classification of chemical sensors, descriptions of chemical sensors (electrochemical sensors, potentiometric sensors, Volta-metric chemical sensors, sensors based on conducting properties), Optical sensors (light guides, the evanescent wave, design of fibre optic sensor, indicator mediated sensor), Calorimetric sensors (catalytic gas sensor, thermal conductivity sensor), mass sensor (piezoelectric quartz crystal resonator, surface acoustic wave sensor).

6. Biosensors in analysis (4H)

Introduction, producing biological surface, methods of immobilization, Achievement of biotransduction (amperometric, potentiometric, optical).

References:

1. Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley –VCH
2. Laboratory automation, Industry 4.0 MOOCs / Swayam Courses

---

Section II : Analytical Chemistry of Agriculture, Polymer and Detergents (Lecture: 30H)

---

1. Analysis of soil (10H)

a) Sampling of soil, sample preparation, Pre-treatment of Samples and Contamination, Trace Element Analysis, Sub-sampling, Drying Techniques, Milling, Grinding and homogenization, c) Acid-digestion, Ashing and Extraction Procedure for soils d) Analysis of Soil: Soil Analytical Procedures - Determination of extractable boron, Cation exchange capacity, exchangeable bases and base Saturation, Determination of CEC and exchangeable cations, Measurement of calcium and magnesium by AAS, Measurement of potassium and sodium by flame photometry, Determination of cation exchange capacity (CEC), Determination of effective cation exchange capacity (ECEC), Determination of fulvic and humic acids, Determination of nitrogen, Determination of soil organic matter, Determination of Sulpur, phosphorus, magnesium, potassium, Sodium and trace elements, Discussion-Determination of extractable sulphur, Analysis of Pesticide Residues

2. Polymer analysis (10H)

Introduction and Types of polymers. Identification methods, Solubility, Density, Behaviour on Heating; Spectroscopic methods, Molecular Weight Calculations, Viscometry, Chromatography, Ultracentrifugation, Osmometry, Light Scattering, End-Group Analysis, Turrbidimetric Titration, Thermal Analysis, Isomerism, Chain Dimensions, Crystallinity, Orientation, Blends, Thermal Behaviour, Dilatometry.

Resonance Spectroscopy, Optical Microscopy, Transmission Electron Microscopy, X-Ray Diffraction, Neutron Scattering,

3. Analysis of Surfactants (10H)

a) Surfactant types; classification, identification, separation: Why analyse surfactants, Features peculiar to surfactant analysis, Definitions, types of surfactants ,Elemental analysis: Extraction of surfactants, Acid-base titration, Determination of weak acids and bases and their salts, Potentiometric titration, Two-phase titration of ionic surfactants with surfactants of opposite charge, Introduction, ISO 2271: , Analysis of Representative surfactants: i) Analysis of Anionics ii) Analysis of nonionics: iii) Analysis of cationics and amphoterics

References:

1. Methods in Agricultural Chemical Analysis: A Practical Handbook, N.T. Faithfull, CABI Publishing, Typeset by Wyvern 21 Ltd, Bristol (2002).
2. Soil Sampling and Methods of Analysis, Edited by M.R. Carter E.G. Gregorich, Canadian Society of Soil Science, Second Edition (2008)

3. Manual of Pesticide Residue Analysis Volume I, Edited by Hans-Peter Thier and Hans Zeumer, Pesticides Commission, VCH, New York.
4. Polymer analysis, Barbara H. Stuart, Analytical Techniques in the Sciences (AnTS), John Wiley and Sons Ltd.
5. Analytical Methods for Polymer Characterization Rui Yang, CRC Press Taylor & Francis Group, 2018
6. Introduction to Surfactant Analysis, Edited by D. C. Cullum, Springer-Science + Business Media, B.V, 1994.
7. Handbook of Detergents, Editor-In-Chief Uri Zoller, Part-C, Heinrich Waldhoff, Rüdiger Spilker, Marcel Dekker, New York, 2005.

PSCHA 243: B) Laboratory Automation and Environmental Analytical Chemistry
(Lecture: 30H)

1.Define / understand various terms in – i) Laboratory automation and sensors, ii) environmental pollution, analysis water and air.
2. Explain instrumentation of automated laboratory analysis and sensors.
3. To describe basic principles of automated laboratory analysis and sensors.
4. Explain importance of automated laboratory analysis and sensors.
5.Describe sources of water and air pollution and pollutants.
6.Describe / explain methods / techniques of sampling of water and air and their analysis.
7. Solve numerical problems on analysis water and air.
8. Draw conclusion regarding water and air quality from analytical results.

Section-I: Laboratory Automation and Sensor Based Techniques (Lectures 30 H)

---

1. Introduction to laboratory Automation (2H)

Introduction, automation, miniaturization and simplification, lab automation, flow injection analysis, miniaturized analytical systems, fast response analytical systems, chemical sensors, screening systems, process on-line systems.

2. Laboratory Automation (6H)

Definition and concept, objective of automation in analytical chemistry, automation of analytical tools and process, automation of preliminary operations, automation of calibration, automation of measuring and transducing of analytical signals, automation of data acquisition and processing, analysers, automated management system, advantages and shortcomings of automated system.

3. Flow Injection Analysis (6 H)

Batch and continuous flow analysis, principles, basic FIA instrumentation, dispersion in FIA, FIA for reproducible and precise sample preparation, FIA system with enzymes, flow injection hydride generation scheme, online sample conditioning, and preconcentration, exploiting the physical dispersion process, FIA gradient technique, Process control, process control analysers.

4. Miniaturized Analytical systems (6H)

Introduction, Concept, theory of miniaturization, microfabrication, silicon and glass micro-matching, polymer replication technology, miniaturized analytical components, sampling and sample pre-treatment, system integration, serial integration, parallel integration, commercialization.

5. Chemical Sensors (6H)

Introduction, definitions, Classification of chemical sensors, descriptions of chemical sensors (electrochemical sensors, potentiometric sensors, Volta-metric chemical sensors, sensors

based on conducting properties), Optical sensors (light guides, the evanescent wave, design of fibre optic sensor, indicator mediated sensor), Calorimetric sensors (catalytic gas sensor, thermal conductivity sensor), mass sensor (piezoelectric quartz crystal resonator, surface acoustic wave sensor).

6. Biosensors in analysis (4H)

Introduction, producing biological surface, methods of immobilization, Achievement of biotransduction (amperometric, potentiometric, optical).

References:

1. Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley –VCH
2. Laboratory automation, Industry 4.0 MOOCs / Swayam Courses

---

Section-II: Environmental Analytical Chemistry (Lectures 30 H)

---

1. Water Pollution and Measurement of Water Quality (16 H)

a) Water Pollutants: Brief explanation of following with respect to their sources and toxic effects - Inorganic pollutants (Heavy Metals (CD, Hg, Pb), Metalloids, Organotin Compounds, Inorganic Species (CN-, NH3 and other species), Asbestos), Organic Pollutants (Soaps, Detergents, and Detergent Builders, Pesticides in Water, Polychlorinated Biphenyls), Emerging Water Pollutants, Pharmaceuticals, and Household Wastes, Radionuclides in the Aquatic Environment).

b) Analysis: Physical Properties: Colour (Visible Inspection, Spectrophotometric—Multi-Wavelength Method, Turbidity, Odour, Taste, Acidity, Alkalinity, Calcium Carbonate Saturation, (Introduction, Indices Indicating A Water's Tendency To Precipitate Or Dissolve Caco3, Indices Predicting The Quantity Of CaCO3 That Can Be Precipitated Or Dissolved), Hardness, Oxidant Demand/Requirement (Chlorine Demand/Requirement, Ozone Demand/Requirement— Batch Method), Conductivity, Salinity. Metal ions: Introduction, Preliminary Treatment Of Samples (Introduction, Filtration for Dissolved and Suspended Metals, Treatment for Acid-Extractable Metals, Digestion for Metals, Nitric Acid Digestion, Nitric Acid-Hydrochloric Acid Digestion, Nitric Acid-Sulfuric Acid Digestion, Nitric Acid-Perchloric Acid Digestion, Nitric Acid-Perchloric Acid Hydrofluoric Acid Digestion, Dry Ashing, Microwave-Assisted Digestion), Quantitative analysis by AAS, FES and ICPAES: Only general explanation as this part is covered in detail in Analytical spectroscopy Sec-I. c) Inorganic non-metal: Introduction, Determination of Anions By Ion Chromatography, Inorganic Anions By Capillary Ion Electrophoresis; Bromide (phenol red method), cyanide, Chlorine (DPD colorimetric method), Fluoride (ion selective method, complexone method), ammonia (titrimetric method, ions elective method and phenate method), NO_2^- - colorimetric method, NO_3- (nitrate electrode and Cd reduction method), Organic nitrogen by Microkjeldahl method, Dissolved oxygen (iodometric and membrane electrode method), phosphate (molybadate – $SnCl_2$ - colorimetric method), Sulfide (methylene blue and ion selective method), d) Organic constituents: Biochemical oxygen demand, Chemical oxygen demand, total organic carbon, phenols (direct photometric method), surfactants.

2. Air Pollutants and Analysis of the Atmosphere and Air Pollutants (14H)

a) Air Pollutants: Explanation only with respect to source and health hazards of: CO, SO_2, NOx, NH_3, Cl_2 and F_2; Organic Pollutants (Aromatic Hydrocarbons, Carbonyl Compounds, Miscellaneous Oxygen-Containing Compounds, Organonitrogen Compounds, Organohalide Compounds, Organosulfur Compounds, Organic Particulate Matter, Hazardous Air Pollutants Organic Compounds) (Ref-2: 285 to 329 only relevant information from these pages)

b) Pollutant Analysis: Atmospheric Monitoring, Air Pollutants Measured, Sampling, Methods of Analysis, determination of Sulfur Dioxide, Nitrogen Oxides, Analysis of Oxidants, Contents,

Analysis of Carbon Monoxide, Determination of Hydrocarbons and Organics, Determination of Specific Organics in the Atmosphere, Analysis of Particulate Matter, Filtration, Collection by Impactors, Particle Analysis, X-Ray Fluorescence, Determination of Lead in Particulate Matter, Direct Spectrophotometric Analysis of Gaseous Air Pollutants.

References

1. Standard methods for the examination of water and waste wter, 23rd Ed. Rodger Baird, Andrew Eatson, Eugene Rice, jointly published by: Americal Public Health Association, Americal Water Works Association, Water Environment Fedearation.
2. Environmental Chemistry, Stanley E. Manahan, Ninth Edition, CRC press, Taylor and Francis, 2010.
3. Handbook of Environmental Analysis Chemical Pollutants in Air, Water, Soil, and Solid Wastes by Pradyot Patnaik, Third Edition, CRC press, Taylor and Fransis, 2018.
4. Environmental Chemistry, A. K. Day, New Age Publication Company

---

PSCHA 244: Analytical Chemistry Practical II (4 Credit- Hours)

---

1. Maintain proper record of analytical data in notebook. Observer personal safety in laboratory and able handle all chemicals, instruments, etc safely in laboratory.
2. Define / understand various terms involved practical methods of quantitative analysis.
2. To analyse organic and inorganic materials using appropriate chemical / instrumental methods
3. Explain / describe basic principles of chemical / instrumental methods used for analysis.
4. Perform analysis of sample with described procedure
5. Apply / select particular method / instrumental parameters for analysis of given sample.
6. Maintain appropriate reaction conditions as described in procedures.
8. To conclude the results able to take the decision regarding quality of sample.
9. To perform calculations and interpret the results.

Section I : Any 12 experiments

1.Table Work: Characterization of organic compounds by VU-Visible, IR and NMR spectroscopy (any two compounds, Example- paracetamol and aspirin -actual spectra must be given for analysis)

2.Analytical Chemistry for Self-Employment: Preparation / Isolations Analytical Standards or reference material for analytical laboratories (Note: all these materials can be used for further experiments)

3.Solvent extraction: Isolation and purification caffeine. Impurity present if any by TLC. Indian Pharmacopeia Tests: identification tests, MP, loss on drying, Total heavy metal and assay. (Spectral characterization may be performed) (Ref-5)

4-5. Synthesis of Paracetamol (or any other medicinal compound) by green chemistry route and recrystallization. Test as per IP: TLC, MP, Identification tests, limit test for chloride, LOD and assay. (spectral characterization may be performed) (Ref-5 and 4)

6. Analysis of creatinine (trinitrophenol method) (Ref-3)

7. Blood cholesterol (ferric chloride method) (Ref-3)

8. Analysis of proteins by Lowry method (Ref-3, 6)

9. Analysis of reducing sugar by colorimetry method. (Ref-3, 6)

10. Limit tests for heavy metal to Aurvedic medicinal preparations

11. Synthesis of Methyl red indicator, purification, MP/ TLC and test for colour change with respect to change in pH of indicator,

12. Identification of amino acids / sugars / or any other mixture by two-dimensional chromatographic method (TLC or paper) (Ref.-3)

13. Analysis of protein or DNA by gel electrophoresis

14. Determination of molecular weight by gel permission column chromatography (Sephacryl S-200 column) (Ref-6)
15.Separation of amino acids by ion exchange chromatography (Ref-6)
16.Determine amount of magnesium from given talcum powder

Self activity

1.Preparation of Analytical Reagent Kit (any two) which will contain all the reagents for determination of specific analyte, labelling and packaging of reagents and writing of standard protocol to use the kit (such kits are used in commercial analytical laboratories)

Section – II: Any 12 experiments

---

1. Analysis of Cement SiO_2, Calcium, Iron, Magnesium and Aluminium (Ref-1)
2.Analysis of mixed fertilizer sample for total nitrogen, K and phosphate content. (Ref-1)
3.Analysis of dolomite ore with respect to SiO_2, Ca and Mg (Ref-1)
4.Analysis of brass alloy for Cu and Sn (Ref-1)
5.Determination of total Ash, Ash Insoluble in Hydrochloric acid, Alkalinity of soluble ash
6.Use of ion selective electrodes for determination (F, Cl, Ca, NH4+ etc. from water)
7.Determination of Ca in milk powder by flame photometry by standard addition or calibration curve method (FSSAI Manual)
8.Determination of molecular weight by gel permission column chromatography (Sephacryl S-200 column) (Ref-6)
9. Spectrophotometric determination of lead in leaves using dithizone-chelating agent
10. Analysis of Salbutal sulphate from asthma inhaler by UV Spectrophotometry
11. Removal of dyes on activated charcoal by column chromatography
12. Determination of COD from waste water
13. Determination of calcium from given sample of plaster of Paris
14. To determine phosphoric acid in cold drink by molybdenum blue method.
15.Detection of amino acids by ninhydrin after thin layer chromatographic Separation
16.Moisture content by i) Loss on drying of caffeine (oven drying method) and water content of dextrose (anhydrous or monohydrate) by Karl Fischer Method. (Ref-1 and 2)
17.Kit method (any two): a) Analysis of glucose from blood or hydrolysed food sample and b) urea from urine, c) Cholesterol from blood or fatty material. d) Creatinine
Determination anionic detergents from waste water (artificially prepared water sample containing detergent or shampoo which contain sodium lauryl sulphate or ammonium lauryl sulphate) (Ref-1, 2, 3)
18. Photochemical remediation of pollutants (Ref-1)
Chemical mineralization of pollutants by Fenton's Process (Ref-1)
19. Molecular weight of polymer by viscometer

PSCHAP-245: Project (4 Credit- 60 Hours)

a) All students of M. Sc.-II must be allotted projects
b) Each student will perform project separately. The project resort should consist of title page, certificate, table of content, summary of project followed by introduction, literature survey, experimental methods, results, discussion and conclusions. Report may also include Appendices consisting of 1) references, 2) standard spectra / data if any and 3) safety precautions. If student is willing to do project in another institute, internal mentor must be allotted and he will be responsible for internal assessment of a student. In this case student has to obtain certificate from both external and internal supervisor. Systematic record of attendance of project students must be maintained by a mentor. Project will be evaluated

jointly by three examiners and there will not be any practical performance during the examination. Progress of project may be carried out mid semester presentation.
