Savitribai Phule Pune University

(Formerly University of Pune)

Two Year Post-Graduate Program in Chemistry

(Faculty of Science & Technology)

Choice Based Credit System Syllabus-2023

According to NEP-2020

M.Sc. Part-II

Analytical Chemistry

for

Colleges Affiliated to Savitribai Phule Pune University

Implementation from Academic Year

2024 - 2025

M. Sc.-II Analytical Course Structure

Semester-III				
Major Compulsory				
1	Major Theory Paper-1	CHA-601 MJ	Major Core	4C-T
2	Major Theory Paper-2	CHA-602 MJ	Major Core	4C-T
3	Major Theory Paper-3	CHA-603 MJ	Major Core	2C-T
4	Major Practical paper-1	CHA-604 MJP	Major Core	2C-P
5	Major Practical paper-2	CHA-605 MJP	Major Core	2C-P
Major Elective (any one theory and any one practical paper)				
6	Minor Theory Paper-1	CHA-606(A) MJ	Major Elective Theory	2C-T
	Minor Theory Paper-2	CHA-606(B) MJ		2C-T
	Minor Theory Paper-3	CHA-606(C) MJ		2C-T
7	Minor Practical paper-1	CHA-607(A) MJP	Major Elective practical	2C-P
	Minor Practical paper-2	CHA-607(B) MJP		2C-P
Research Project Compulsory				
8	Research Project (RP)-1	CHA-608 RP	Research Project	4C-P
Semester-IV				
Major Compulsory				
1	Major Theory Paper-4	CHA-651 MJ	Major Core	4C-T
2	Major Theory Paper-5	CHA-652 MJ	Major Core	4C-T
3	Major Theory Paper-6	CHA-653 MJ	Major Core	2C-T
4	Major Practical Paper-7	CHA-654 MJP	Major Core	2C-P
Major Elective (any one theory and any one practical paper)				
6	Minor Theory Paper-4	CHA-655(A) MJ	Major elective Theory	4C-T
	Minor Theory Paper-5	CHA-655(B) MJ		4C-T
	Minor Theory Paper-6	CHA-655(C) MJ		4C-T
7	Minor Practical paper-3	CHA-656(A) MJP	Major elective Practical	2C-P
	Minor Practical paper-4	CHA-656(B) MJP		2C-P
Research Project Compulsory				
6	Research Project (RP)-2	CHA-657 RP	Research Project	6C-P

M. Sc.-II Analytical Chemistry Paper Description

SEMESTER-III				
Major Compulsory				
1	Thermal and Extraction Techniques in Analytical Chemistry	**CHA-601 MJ**	Major Core	4C-T
2	Advanced Chromatographic Method of Chemical Analysis	**CHA-602 MJ**	Major Core	4C-T
3	Applied Electro-analytical Techniques	**CHA-603 MJ**	Major Core	2C-T
4	Practical: Advanced Instrumental Methods of Chemical Analysis	**CHA-604 MJP**	Major Core	2C-P
5	Practical: Analytical Method Development	**CHA-605 MJP**	Major Core	2C-P
Major Elective: any one theory + any one practical				
6	Analytical Methods to Examining Water and Soil Quality	**CHA-606(A) MJ**	Major elective theory	2C-T
	Automation and Sensor in Analytical Chemistry	**CHA-606(B) MJ**		
	Forensic Analytical Chemistry	**CHA-606(C) MJ**		
7	Practical: Measuring Water and Soil Quality	**CHA-607(A) MJP**	Major elective Practical	2C-P
	Practical Forensic Chemistry	**CHA-607(B) MJP**		
Research Project Compulsory				
8	Research Project (RP)	**CHA-608 RP**	Research Project	4C-P
SEMETER-IV				
Major Compulsory				
1	Applied Analytical Spectroscopy	**CHA-651 MJ**	Major Core	4C-T
2	Chemical Methods of Pharmaceutical Quality Control	**CHA-652 MJ**	Major Core	4C-T
3	Bio-Analytical Techniques	**CHA-653 MJ**	Major Core	2C-T
4	Practical: Pharmaceutical Analysis for Quality Control	**CHA-654 MJP**	Major Core	2C-P
Major Elective: any one theory + any one practical				
5	Analytical Methods of Food Quality Control	**CHA-655(A) MJ**	Major elective Theory	2C-T
	Clinical Analytical Chemistry	**CHA-655(B) MJ**		
	Analytical Techniques For Polymer Characterization	**CHA-655(C) MJ**		
6	Practical: Methods of Food Quality Determination	**CHA-656(A) MJP**	Major elective Practical	2C-P
	Practical Clinical Biochemistry	**CHA-656(B) MJP**		
Research Project Compulsory				
7	Research Project (RP)	**CHA-657 RP**	Research Project	6C-T

M. Sc.-II: Analytical Chemistry - Program Outcomes

PO-1: Disciplinary knowledge and skill: A post graduate student is expected to be capable of demonstrating comprehensive knowledge and understanding both theoretical and practical fundamental concepts in all disciplines of Analytical Chemistry. Further, the student will be capable of applying modern technologies, handling advanced instruments and Analytical Chemistry related soft-wares for chemical analysis, characterization of materials and in separation technology.

PO-2: Skilled communicator: The course curriculum incorporates basics and advanced training in order to make a post graduate student capable of expressing the subject through technical writing as well as through oral presentation.

PO-3: Critical thinker and problem solver: The course curriculum also includes components that can be helpful to post graduate students to develop critical thinking and to design, carry out, record and analyze the results of chemical reactions. Students will be able to think and apply evidence based comparative chemistry approach to explain chemical analysis and reactions / mechanism involved in it.

PO-4: Sense of inquiry: It is expected that the course curriculum will develop an inquisitive characteristic among the students through appropriate questions, planning and reporting experimental investigation.

PO-5: Team player: The course curriculum has been designed to provide opportunity to act as team player by contributing in laboratory, field-based situation and industry.

PO-6: Skilled project member / manager: The course curriculum has been designed in such a manner as to enabling a post-graduate student to become a skilled project manager by acquiring knowledge about chemistry project management, writing, planning, study of ethical standards and rules and regulations pertaining to scientific project operation.

PO-7: Digitally literate: The course curriculum has been so designed to impart a good working knowledge in understanding and carrying out data analysis, use of library search tools, use of chemical simulation software and related computational work.

PO-8: Ethical awareness: A post graduate student requires understanding and developing ethical awareness or reasoning which is adequately provided through the course curriculum.

PO-9: Environmental Awareness: As an inhabitant of this green planet Analytical Chemistry post graduate student should have many social responsibilities. The course curriculum is designed to teach Analytical Chemistry post graduate student to follow the green routes for the synthesis of chemical compounds and also find out new greener routes for sustainable development. The course also helps them to understand the causes of environmental pollution and thereby applying environmentally friendly policies instead of environmentally hazard ones in every aspect.

PO-10: Analytical skill development and job opportunity: The course curriculum is designed in such a way that Analytical Chemistry graduate students can handle many Chemistry based software, modern instruments and advanced technologies to synthesize, characterize and analyze the chemical compounds very skillfully. Such a wonderful practice in the graduate level will bring a good opportunity to the students for getting job in industries besides academic and administrative works.

Paper-wise Syllabus, SMESTER – III

CHA-601 MJ: Thermal and Extraction Techniques in Analytical Chemistry

[Compulsory Theory Paper, Four credits] [60 L]

Section-I: Thermal Methods of Analysis

1. Introduction to Thermal Methods [3 L]

Introduction, Historical development, Definitions: Thermal analysis, Equilibrium -A Kinetic Diversion, General apparatus, Factors affecting thermal analysis results, The sample, The crucible, The rate of heating, The atmosphere, The mass of the sample, Simultaneous and complementary techniques
(Ref-1: 1-21)

2. Thermogravimetry [6 L]

Introduction, Historical, Definition of thermogravimetry, Apparatus, The balance, Furnace, Programmer, Samples, Temperature calibration, Atmosphere, Kinetics of reactions, Kinetics of Reactions, Measurement of α and dα/dt, Constant rate methods, Thermogravimetric curves: Decomposition Of Magnesium Hydroxide, Calcium oxalate monohydrate, Copper sulphate pentahydrate, Degradation of polymers, Analysis of mixtures: mixtures of alkaline earth oxalates, polymer blends, soils, Oxidation studies, Reduction studies, Controlled rate thermogravimetry and Hi-Res ™ TGA, Polymer blends, Drugs.
(Ref-1:22 to 62)

3. Differential Thermal Analysis and Differential Scanning Calorimetry [8 L]

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 DT A and DSC, Heat flux DSC, Power-compensated DSC, The effect of higher temperatures, Sample size, Calibration, Applications: Physical changes and measurements (crystalline phase transitions, potassium nitrate, liquid crystalline transitions, thermoplastic polymer phase changes, heat capacity measurements, glass transition temperatures), Chemical reactions, Inorganic compounds and complexes (calcium oxalate monohydrate, metal complexes, high alumina cements, clays and other minerals), Organic compounds (oxidative degradation, protein denaturation, polymer degradation).
(Ref-1: 63-113)

4. Thermomechanical and Dynamic Mechanical Analysis [5 L]

Introduction, Definitions: Thermomechanical analysis, Dynamic mechanical analysis, Mechanical moduli, Thermomechanical analysis: Apparatus (probes, calibration),Applications: coefficients of expansion, solvent swelling of polymers, phase transitions, sintering), Chemical reactions (inorganic hydrates, polymer cure), Dynamic Mechanical Analysis: Apparatus (DMA configurations, calibration) Applications: glass transition temperatures, beta and other transitions, relaxation kinetics, polymer miscibility, characterising cross-linking, studying 'problem samples, characterising film formation
(Ref-1: 123-151)

5. Simultaneous Techniques and Product Analysis [5 L]

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.
(Ref-1: 163-184)

6. Problem Solving and Applications of Thermal Methods [3 L]

Introduction, List of examples, Problems: Inorganic materials, Polymeric materials, Fine chemicals and pharmaceuticals, other materials, Solutions to problems. (Ref-1: 206-270) (This topic is for student's self-preparation)

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.

Section-II: Analytical Extraction Techniques

1. Pre and Post Extraction Consideration [2 L]

Organic compounds of interest, pre-sampling issues, sampling strategies-solid, aqueous and air samples, chromatographic method of analysis, sample preconcentration methods. (Ref-1: 1-29)

1. Classical Approach for Aqueous Extraction [6 L]

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, synergic extraction, extraction reagents for metals, selection of solvents, solvent extraction, problems with LLE process), purge and trap for volatile organics in aqueous samples, Examples of Solvent Extraction- estimation individual metal ions Be, B, Cu, Fe and Pb by solvent extraction. Problems. (Ref-2: Relevant pages and Supplementary Ref.-1: 39-45)

2. Solid Phase extraction (SPE) [8 L]

Introduction, Types of SPE media, SPE formats and apparatus, method for SPE operation, solvent selection, factors affecting SPE, selected methods of analysis for SPE: application of normal phase SPE, application of reversed phase SPE, application of ion exchange SPE, applications of molecularly impaired polymers, Automation and On-Line SPE and its applications. (Ref-1: 49-78)

3. Solid phase micro-extraction [8 L]

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: analysis of abitic dehydroabietic acid in food samples, analysis of fungicide in water. Automation of SPME and its application, New development in micro extraction (Introduction, stir bar sorptive extraction, liquid phase micro-extraction, , membrane micro extraction, micro extraction in packed syringe. (Ref-1: 85-110, Ref-3)

4. Solid -Liquid Extraction, Microwave extraction [6 L]

Classical Approach: Introduction, Soxhlet extraction, Automated Soxhlet extraction, other approaches, Pressurized Fluid Extraction: Introduction, theoretical consideration, Instrumentation

for PFE, method development and applications. Microwave assisted extraction: Introduction, instrumentation, Applications. (Ref-1: 125-174)

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 Micro-extraction, A Practical Guide, Edited by Sue Ann Scheppers Wercinski, CRC press, Taylor and Francis.

Course Outcomes - At the end of course, students should able to

CO-1 Define/ understand various terms used in thermogravimetry and analytical extraction techniques.
CO-2 Explain instrumentation in thermal methods and extraction techniques.
CO-3 Describe basic principles of thermogravimetry and extraction techniques
CO-4 Explain /Describe applications of thermal methods and extraction techniques in industry and in analytical laboratory
CO-5 Apply / select particular method of analysis for sample to be analysed.
CO-6 Solve numerical problems on thermal methods of analysis and Extraction
CO-7 Interpret, thermogram, differential thermogram and DSC thermogram.
CO-8 Differentiate among the various methods of thermal analysis and extraction techniques

---

CHA-602 MJ: Advanced Chromatographic Method of Chemical Analysis

[Compulsory Theory Paper, Four Credits] [60 L]

Section-I: Gas Chromatography and GC-MS

1. Fundamentals of Chromatographic Methods of Analysis [5 L]

Fundamentals of Chromatographic Separation (overview, the development of chromatogram), Characteristics value in chromatogram, Chromatographic theories (plate theory, kinetic theory), R_s as measure of peak separation, qualitative and quantitative analysis. Problems. ***(Ref-2,*** *Supplementary Ref-1, 6)*

2. Gas Chromatography [7 L]

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.***(Ref.-2,*** *Supplementary Ref-1, 6)*

3: Super Critical Fluid Chromatography and Extraction [4 L]

Properties of supercritical fluid, Supercritical fluid chromatography: Instrumentation and operating variables, effect of pressure, stationary phases, mobile phases, detectors, comparison with other types of chromatography, Applications, supercritical fluid extraction: Advantages of SFE, instrumentation, of line and on line extraction, applications.**(Ref-4:** 856-865,supplementary Ref-1*)*

4. Gas Chromatography-Mass Spectrometry [10 L]

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.***(Ref-1:** 79-134)*

5. Applications of GC and GC-MS [4 L]

1. Quantitative analysis by GLC-different methods, Elemental Analysis using Gas Chromatography, analysis of Al, analysis of a mixture using the interna1 normalisation method, determination of sucrose as its trimethylsilyl derivative using gas-liquid chromatography, ***Ref-4***
2. Phenols in waste water by LLE-GC method *(sec-6420 phenols)*, Organochlorine pesticides in water: LLEGC method-1, LLEGC method-2 *(sec-6630 organochlorine pesticides)*, volatile organic compounds – Purge and trap capillary column GC-MS method *(Sec-6200-A,B,C)*, Tributyl tin by GC-MS and FID method *(Sec-6710-A,B,C)* ***Ref- 5***

References

1. Basic Gas Chromatography Mass Spectrometry, Principles and Techniques, F.W. Karasekand R.E. Clement, Elsevier, (Elsevier Science B.V.) 1988
2. Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley – VCH
4. Vogels's, Textbook of Quantitative Chemical Analysis 6th Ed.
5. 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,
6. Forensic applications of Gas Chromatography by Michelle Carlin and John Dean, CRC press, 2013)

Section-II: Liquid Chromatography

1. Instrumentation of HPLC [6 L]

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*; Columns and Stationary Phases: *Columns for HPLC, Precolumn, General properties of stationary phases, Silica, Chemically modified silica, Styrene-divinylbenzene, Column care and regeneration(**Ref-2:** 1-9, 59-136, Ref-1)*

2. HPLC Methods [8 L]

a) Adsorption Chromatography: Normal-Phase Chromatography: What is adsorption?, The eluotropic series, Selectivity properties of the mobile phase,Choice and optimization of the mobile phase, Applications*(**Ref.-2:** 159-168, Ref-1)*

b) Reversed-Phase Chromatography: Principle, Mobile phases in reversed-phase chromatography, Solvent selectivity and strength, Stationary phases, Method development in reversed-phase chromatography, Applications, Hydrophobic interaction chromatography. ***(Ref.-2:** 173-191, Ref-1)*

c) Chromatography with Chemically Bonded Phases: Introduction, Properties of some stationary phases, Hydrophilic interaction chromatography, ***(Ref.-2:** 195-200, Ref-1)*

d) Ion-Exchange Chromatography: Introduction, Principle, Properties of ion exchangers, Influence of the mobile phase, Special possibilities of ion exchange, Practical hints, Applications ***(Ref.-2:** 203-213, Ref-1)*

e) Ion-Pair Chromatography: Introduction, Ion-pair chromatography in practice, Applications ***(Ref.-2:** 217-221, Ref-1)*

f) Ion Chromatography: Principle, Suppression techniques, Phase systems, Applications ***(Ref.-2:** 225-230, Ref-1)*

g) Size-Exclusion Chromatography: Principle, The calibration chromatogram, Molecular mass determination by means of size-exclusion chromatography, Coupled size-exclusion columns, Phase systems, Applications. ***(Ref.-2:** 231-244, Ref-1)*

h) Affinity Chromatography: Principle, Affinity chromatography as a special case of HPLC, Applications. ***(Ref.-2:** 249-252)*

3. Analytical HPLC [3 L]

Qualitative analysis, Trace analysis, Quantitative analysis, Recovery, Peak-height and peak-area determination for quantitative analysis, Integration errors, The detection wavelength, Derivatization, Unexpected peaks: Ghost and system peaks. ***(Ref.-2:** 285-308)*

4. Separation of Enantiomers [3 L]

Introduction, Chiral mobile phases, Chiral liquid stationary phases, Chiral solid stationary phases, Indirect separation of enantiomers. ***(Ref.-2:** 333-345)*

5. Mass Spectrometry, LCMS Interface and applications [10 L]

Interface Technology: Introduction, Thermo-spray interface, The electron spray interface (mechanism of electron-spray ionization, sample types, the electro-spray spectrum, structural information from electrospray ionization), atmospheric pressure chemical ionization interface and the mechanism of atmospheric pressure chemical ionization. Data acquisition (identification, quantitation-selected ion monitoring), Processing of mass spectra (total ion current trace, qualitative analysis, quantitative analysis). **Applications:** Molecular weight determination of small molecules (Method Development for Structural Studies, The Use of Target-Compound Analysis and LC–MS–MS for the Identification of Drug Metabolites, The Use of High-Accuracy Mass Measurements in Combination with LC–MS for the Structure Determination of Drug Metabolites, The Use of Cone-Voltage Fragmentation in Conjunction with High-Accuracy Mass Measurements and LC–MS for Metabolite Identification, The Use of LC–MS^n for the Identification of Drug Metabolites),Quantitation (requirements, quantitative standardization, matrix effect in LC-MS, the method of standard addition to overcome matrix effect). ***(Ref-3:** 75, 94-123, 189-218)*

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.

Course Outcome - At the end of course students should able to-

CO-1. Define / understand various terms in chromatography (GC and HPLC) and mass spectroscopy.

CO-2. Explain instrumentations in chromatography (GC and HPLC) and mass spectroscopy.

CO-3. Explain / describe i) basic principles of chromatography (GC and HPLC) and mass spectroscopy. ii) separation in GC / HPLC column. iii) Functioning and construction of GC / HPLC/ MS detectors.

CO-4. Explain /Describe applications chromatography (GC and HPLC) in industry and in analytical laboratory.

CO-5. Apply / select particular method / instrumental parameters for analysis for sample GC / HPLC.

CO-6. Solve numerical problems on chromatography (GC and HPLC) and mass spectroscopy.

CO-7. Integrate GC and HPLC chromatogram, Mass spectrum

CO-8. Differentiate among the chromatography (GC and HPLC) methods of analysis.

CHA-603 MJ: Applied Electro-analytical Techniques

[Compulsory Theory Paper, Two Credits] [30 L]

1. Potentiometry with ion selective electrodes [6 L]

General principles, Definitions: Reference electrode, working electrode, electrode and standard electrode potential, Reference electrode (saturated calomel and Ag-AgCl electrode), Membrane indicator electrodes, Classification of membranes, glass electrode for pH measurement, crystalline membrane electrodes, Liquid membrane electrodes, Ion selective FET, Molecular selective electrode systems (gas sensing probes, Biosensors), instrument for measuring cell potential, Direct potentiometric measurement, Potentiometric titrations (***Ref-1:*** *649 to 682, relevant part only*).

2. Polarographic Methods of Analysis [15 L]

Fundamental principle of Voltammetric analysis and electrode system for voltammetric analysis; Fundamental principle of Polarographic analysis and electrode system for polarographic analysis; Classification of Voltammetric Methods; Principle of Polarographic Analysis; Working Electrode in Polarographic Analysis; Dropping mercury electrode (DME); Construction and working Dropping mercury electrode (DME);Advantages of DME; Disadvantages of DME; Apparatus for polarographic analysis; Instrumentation of Polarography; Electrode system for polarographic cell containing analyte; System for removal of dissolve oxygen; Electrical circuit for application of linear DC potential; Recorder for obtaining polarogram; Working of Polarography; Qualitative and Quantitative Analysis by polarogram; Factors Affecting the Nature of the Polarographic Wave; Residual current; Migration current; Condenser or non-faradic current; Diffusion or faradic current; Half wave potential; Applications of Polarography: Qualitative analysis/Identification of the electroactive species; Quantitative analysis by Ilkovic equation; Simultaneous determination of cations in the mixture; Polarographic determination of copper and

zinc in the brass; Polarographic titration; Determination of dissolved oxygen; Analysis of organic compounds

3. Hydrodynamic Voltametry [03 L]

Hydrodynamic voltametry and applications of hydrodynamic voltametry (voltammetric detectors in chromatography and flow injection analysis, Voltametric oxygen sensors, amperometric titration, problems

4. Cyclic Voltametry [03 L]

Principle of cyclic Voltammetry, cyclic voltamogram of K3[Fe(CN)6] and parathion (*Fundamental studies*), determination of analytes using cyclic voltammetry, criteria of reversibility of electrochemical reactions, quasi-reversible and irreversible processes

5. Stripping Voltammetry [03 L]

Stripping voltammetry, Principle, Electrode System, Anodic Stripping Voltametry and Cathodic Stripping Voltammetry, adsorptive striping voltammetry and their applications, problems

6. Pulse Polarography [02 L]

Different types of excitation signals in pulse polarography, Differential pulse polarography, square wave polarography, Voltammetry with ultra-microelectrode, Applications of these techniques for determination Cu and Zn from tap water by differential pulse polarography and by square wave polarography, Vitamin-C by differential pulse polarography, Determination of Pb in tap water by stripping method, Problems

References

1. ***'Textbook of Quantitative Chemical Analysis',*** Jeffery G. H., Bassett J., Mendham J., Denney R.C., 5th edition., ELBS London.

2. ***'Principles of Instrumental Analysis'***, 6th Ed., Skoog, Holler and Crouch, Brooks/Cole, Thomson Learning.

3. ***'Instrumental Methods of Analysis'***, 6th Ed., H. H. Willard. L. L. Merritt, J. A. Dean, and F. A. Settle, Jr., CBS Publishing Company

4. ***'Introduction to Instrumental Analysis'*** by R. D. Braun, Pharmamed Press.

5. **'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

6. ***'Cyclic Voltammetry'***, Simultaneous Analysis and Reaction Mechanism, David K Gosser, VCH, 1994.

Course Outcome - At the end of course students should able to-

CO-1. Define / understand various terms in Electrochemistry

CO-2. Explain instrumentations and functioning of polarography, potentiometry, cyclic voltammetry, Stripping methods, and hydrodynamic voltammetry.

CO-3. Explain basic principles of polarography, potentiometry, cyclic voltammetry, Stripping methods, and hydrodynamic voltammetry.

CO-4. Describe applications polarography, potentiometry, cyclic voltammetry, Stripping methods, and hydrodynamic voltammetry.

CO-5. Apply / select particular method / instrumental parameters for analysis for sample polarography, potentiometry, cyclic voltammetry, Stripping methods, hydrodynamic voltammetry.

CO-6. Solve numerical problems on electrochemistry.

CHA-604 MJP: Advanced Instrumental Methods of Chemical Analysis

[Compulsory Practical Paper, Two Credits] [60 L]

Any 12 Experiments

Extraction and Absorbance Spectroscopy

1. Quantitative analysis of Caffeine for tea powder by solvent extraction, its purity by UV-Visible spectroscopy or by FTIR spectroscopy. *(Ref-7)*

2. Quantitative analysis of carotenoids from spinach / lycopene from tomato by solvent extraction. TLC separation to find out number of carotenoids. *(Ref-6, 15)*

3. **Solid Phase Extraction:** Isolation of amino acids from aqueous sample using ion exchange resin and their identification by colorimetric test (very dilute glycine solution can be used as an example of alfa amino acid) *(Ref. 5)* **Or** Isolation of beta carotene from spinach leaves on silica gel cartridge by solid phase extraction and its quantification visible spectrophotometry. *(Ref-7)*

4. **Pre-concentration using solid phase extraction on ion exchange cartridge and estimation.** You can any choose any metal ion which is present below detection limit. You will do preconcentration using ion exchange resin and will estimate by AAS or aqueous colorimetry (not solvent extraction). Example: Preconcentration of Cu(II) from brine (one can use aqueous solution of Cu(II) solution with less than 0.5 ppm conc.) and its estimation using R-Nitroso salt *(Ref-1, 4)*

Flame photometry

5. Flame photometric analysis of water /soil sample for Na^+ and K^+ by calibration curve method (give regression analysis for both curves) *(Ref-1).*

6 .Estimation of K^+ from soil/water sample by standard addition method (give regression analysis of both curves) *(Ref-1).*

Methods of Trace Analysis of metals: Atomic Absorption Spectroscopy

7.**Demonstration Practical by Mentor:** Handling of AAS and study on any metal ion estimation by AAS method with respect to 1) Effect of oxidant to fuel ratio on absorbance, ii) detection limit and iii) linearity range for calibration curve method. (give regression analysis) iv) Effect of other metal ion and absorbance of analyte. *(Ref-1, 15)*

8. Estimation of any two-metal ion by atomic absorption spectroscopy from soil or micronutrient supplement or food sample by calibration curve method. *(Ref-1, 15)*

Turbidimetry / Nephelometry

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). *(Ref-1)*

Photoflurimetry

11. Estimation of quinine sulphate from tablet by calibration curve and its confirmation by standard addition method. *(Ref-1)*

12. Estimation of riboflavin calibration curve and its confirmation by standard addition method. *(Ref-1)*

Polarimetry

13. a) Determination of optical rotation thereby calculate specific rotation of dextrose (glucose) and sugar (sucrose). Express purity of glucose and sugar samples on the basis of specific rotation. (Ref-2) b) Determination of glucose supplement sample by polarimeter. *(Ref-2)*

Quantitative TLC

14. Separation of Colours by TLC / Paper chromatography, their isolation by elution from paper or TLC and quantification by colorimetry. *(Ref-1)*

15. Analysis of the Composition of a Mixture of Nitroanilines by Thin-Layer Chromatography and Ultraviolet/Visible Spectrometry *(Ref.-8)*

HPLC

16. **Demonstration Practical by Mentor**i. Handling of HPLC equipment, choice of mobile phase and column, sample preparation.

ii. Record the chromatogram of pure substance and study a) Effect of conc. on peak area and peak height b) from retention time and length of column calculate number theoretical plates from. c) Qualitative analysis – spiking method and by using retention time d) Quantitative analysis by comparing peak height of sample with standard as well as by comparing peak area of sample with standard.*(Ref.-1, 14, 15)*

17. Estimation of APC tablet by HPLC method *(Ref-1, 3, 8)* or HPLC method developed in your laboratory.

Gas Chromatography

18. **Demonstration Practical by Mentor** Study of GC chromatogram: Record the chromatogram of pure ethanol, acetone, methanol and their mixture. Identify peaks of respective substances in mixture and calculate relative percentage of these three substances by percent area method. Calculate N, resolution of chromatographic column. *(Ref-1)*

19. Analysis of vitamin-A acetate or alfa-tocopherol by GC according to IP method or any other reported method or method developed in your laboratory. *(Ref-2)*

Thermogravimetric Method

20. **Demonstration Practical by Mentor** Study of GC chromatogram: Record the TGA of pure $NaHCO_3$ (room temp to 300 °C). Explain different characteristics of thermogram and quantitative analysis by TGA. Explain how thermal decomposition reaction can be predicted from wt. loss.

21. TGA analysis of dolomite ore for $CaCO_3$ and $MgCO_3$ content *(Ref-1)*

22. TGA analysis $CuSO_4\,5H_2O$ *(Ref-1)*

Cyclic Voltammetry

23.Cyclic voltammetric study of Fe(II)/Fe(III) system. Basic principle and calculation of basic parameters from CV. *(Ref-1, 10, 11)*

24. Quantitative analysis using CV of any one -Vit-C / parathion / nitrobenzene / or any other substance for which your department has developed CV method. (Ref.-12,13).

Potentiometry

25. Construct graphite electrode using graphite rod from the dry pen-cell. Perform redox titration between Fe(II) and $KMnO_4$ using graphite electrode and calomel as reference electrode. Perform same titration using Pt electrode and calomel electrode. Report does Pt can be replaced by graphite or not. Give the reasons.

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. https://chem.libretexts.org/Courses/University_of_California_Davis/UCD_Chem_115_Lab_Manual/Lab_1%3A_Cyclic_Voltammetry
11. Cyclic Voltammetry Experiment James J. Van Benschoten. Jane Y. Lewis, and William R. Heineman, Journal of Chemical Education, Volume 60, Number 9, September 1983 (772-776) and Volume 60 Number 9 September 1983 (702-706)
12. Voltammetric analysis of hydroquinone, ascorbic acid, nitrobenzene and benzyl chloride in aqueous, non-aqueous, micellar and microemulsion media, R. Sripriya M. Chandrasekaran M. Noel, Colloid Polym. Sci (2006) 285: 39–48.
13. Electrochemical Determination of Methyl Parathion using a Modified Electrode, Toxicol. and Environ. Chem., 2003, Vol. 85, Nos. 4–6, pp. 233–241.
14. Analysis of Soft Drinks: UV Spectrophotometry, Liquid Chromatography, and Capillary Electrophoresis, Journal of Chemical Education, Vol. 75 No. 5 May 1998
15. Analytical Chemistry for Technicians, John Kenkel**,** Third Edition, CRC Press LLC, 2003.

Course Outcome – At the end of course students should able to-

CO-1. Maintain proper record of analytical data in notebook. Observer personal safety in laboratory and able handle all chemicals, instruments, etc safely in laboratory.

CO-2. Define / understand various terms involved practical methods of quantitative analysis.

CO-3. Explain instrumentations of colorimeter, spectrophotometer, photoflurometer, TGA, HPLC, GC, Flame-photometer, CV, AAS, etc.

CO-4. Explain / describe basic principles of chromatography different instrumental methods of analysis. Able to handle particular instrument according to SOP.

CO-5. Apply / select particular method / instrumental parameters for analysis of given sample.

CO-6. Verify theoretical principle practically or apply theory to explain practical observations.

CO-7. To conclude the results able to take the decision regarding quality of sample.

CO-8. Differentiate among the various analytical methods / techniques of chemical analysis.

CHA-605 MJP: Analytical Method Development and Validation

[Compulsory Practical Paper, Two Credits] [60 L]

Part-I: Compulsory experiment

1. Table Work: a) Explain with example of each Accuracy, precision, noise level, detection limit, quantitation limit, **b)** Explain with example: Expression of results: Calculation of mean, standard deviation, error and absolute error, significant figure; Propagation of errors c) Explain with example: Calibration curve and standard addition method, Regression analysis of calibration curve and its importance. (Ref-3)

Calculations using EXCEL: mean, standard deviation, plotting calibration curve and equation of line, regression, axis labelling, etc.

2. Table Work: Explain with example – Reliability of results, Confidence interval, Comparison of results (students t test, F-test), comparing the means of two sample, Paired t-test, The number of replicate determinations. Calculations using EXCEL for all these tests using 'statistical' function in EXCEL. (Ref-3).

3-7. Analytical method development and validation (Ref. 1, 6, 7)

Study of visible spectroscopic or colorimetric method for estimation of particular metal ion: **Experiment-1**) **a.** Determination of best pH for colour formation reaction, and determination of λ_{max} for quantitative analysis using limited conc. of metal ion and large excess ligand**;** **Experiment-2) a)** Determination of metal ligand ratio at best pH. B) Detection of possible interfering metal ion (**such as** Ca(II), Mg(II), Mn(II), Ni(II), Co(II), Cu(II), Fe(II)). **Experiment-3)** Estimation of noise level, detection limit, quantisation limit and linearity range (Calculate R^2 value); **Experiment-4)** Estimation of known of metal ion by calibration curve method - validation of method for calibration curve method. (Calculate R^2 value); **Experiment-5**) Estimation of known metal ion conc. at low conc. (near or below detection limit) by standard addition method in triplicate - validation of method for standard addition method. (Calculate R^2 value) (Regression analysis must be performed for both methods and results shall be accepted when R^2 is greater than 0.95)

Metal-Ligand system (any one): Cu(II) - R-nitroso salt; Co(II)-alfa-nitroso beta-napthol; Co(II) - R-nitroso salt; Fe(III) – Salicylic acid.

Note: i) A mentor can practice multiple examples in batch**.** ii) *Student should prepare systematic report in the journal which should contain 1) introduction to UV-Visible spectroscopy, basic terms in absorption spectroscopy, Beer's law, construction and working of colorimeter and spectrophotometer, interpretation of absorbance spectra of organic and inorganic substances, basis of quantitative analysis by UV-Visible spectroscopy, calibration curve method, standard addition method, advantages of*

graphical methods, basis for simultaneous method analysis of non-interfering substance by spectrophotometry. This part will be followed by experiment 3 to 7.

8. Develop the kit for a particular analysis. Prepare reagents, label them, work out the procedure and write validated procedure (Like commercial kit that are available in the market example glucose from blood). **You can choose any example. Few are mentioned here** – 1) Iron from soil-Colorimetry, 2) phosphate from soil-colorimetry 3) Milk adulteration tests, 4) Quality of irrigation water sample, 5) Urea from urine -Colorimetry (**References:** 1-6, 9-11)

Any four experiments from 8 to 13

9. Estimation of Glucose – Glucose in different samples can be analysed by i) titration with Fehling solution b) Colorimetry Folin-Wu method or DNSA method c) Colorimetry-Glucose by oxidase peroxidase method and d) Polarimetry. **Samples are** – a) glucose in saline (DNS), b) glucose in urine / blood sample c) glucose in glucose supplement d) glucose in food. Mentor will assign any one sample to the student. Student have to choice suitable method for analysis of glucose in sample with reason. After discussion with mentor analyse the sample by particular method. **Ref.** – 4, 9, 11.

10. Analysis of Riboflavin sample by calibration curve method by visible spectrometry and Photoflurometry with respect to - linearity range and detection limit. Compare results with respect to sample requirement, detection limit, and accuracy of both methods. Give your choice for analysis of Riboflavin as raw material in pharmaceutical industry *(Ref-4, 6 and 9).*
10. 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. *(Ref-3)*

11. Determine amount of $NaHCO_3$ + Na_2CO_3 from mixture of known composition: **Methods are**
1. Determine amount of $NaHCO_3$ by thermal decomposition method (gravimetry) on burner and calculate amount of both. Determine amount of $NaHCO_3$ + Na_2CO_3 from mixture by volumetric method using standardized 0.05 N HCl. Compare purity or amount of $NaHCO_3$ in sample by both methods. Comment on advantages and disadvantages of each methods. Give your choice of method between two. *(Ref-3)*

12. Perform pH metric titration for estimation of CH_3COOH from vinegar using i) 0.1 M standardized NaOH using phenolphthalein indicator ii) 0.5 M standardized NaOH using pH meter. Compare the results of two methods and give your comment. *(Ref-3)*

13. Determine Paracetamol in tablet conventional titration (redox titration with Ceric ammonium nitrate) and by potentiometric titration (redox titration using Pt and Calomel electrode) and compare the results of two method. *(Ref-3, 6)*

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, 6^{th} Ed, Pearson

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. Manual Of Methods Of Analysis Of Foods Food Safety And Standards Authority Of India Ministry Of Health And Family Welfare Government Of India New Delhi 2015

11. Food Analysis, Edited by S. Suzanne Nielsen, Fourth Edition, Springer.

Course Outcome – At the end of course students should able to-

CO-1. Maintain proper record of analytical data in notebook. Observer personal safety in laboratory and able handle all chemicals, instruments, etc safely in laboratory.

CO-2. Define / understand various chemical terms involved Method development and validation.

CO-3. Explain statistical parameters of Method development.

CO-4. Able to Method development for given analyte.

CO-5. Design / modify and validate new analytical method for chemical analysis of particular sample.

CO-6. Apply / select particular method / instrumental parameters for analysis of given sample.

CO-7. Give mathematical treatment to analytical data and able to interpret the results accurately.

CO-8. To conclude the results able to take the decision regarding quality of sample.

CHA606 (A) MJ: Analytical Methods for Examining Water and Soil Quality

[Optional Theory Paper, Two Credits] [30 L]

1. Water Pollution and Measurement of Water Quality [18 L]

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^-, NH_3 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). (Ref-2: 159-183 supplementary reference-3 and 4)

b) Analysis: Physical Properties: Colour (Visible Inspection, Spectrophotometric—MultiWavelength Method, Turbidity, Odour, Taste, Acidity, Alkalinity, Calcium Carbonate Saturation, (Introduction, Indices Indicating A Water's Tendency To Precipitate Or Dissolve $CaCO_3$, Indices Predicting The Quantity Of $CaCO_3$ That Can Be Precipitated Or Dissolved), Hardness, Oxidant Demand/Requirement (Chlorine Demand/Requirement, Ozone Demand/Requirement— Batch Method), Conductivity, Salinity. (Ref-1: 2.5, 2.8, 2.12-2.40, 2.48-2.62).

c) 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 procedure is expected for methods included here.

(Ref-1: 3.1-3.35, 3.36-3.67, 3.70-3.71, 3.76-3.78, 3.82-3.84, 3.104-3.105).

d) 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 Micro Kjeldahl method, Dissolved oxygen (iodometric and membrane electrode method), phosphate (molybadate – $SnCl_2$ - colorimetric method), Sulfide (methylene blue and ion selective method),

e) Organic constituents: Biochemical oxygen demand, Chemical oxygen demand, total organic carbon, phenols (direct photometric method), surfactants.

(Ref-1: 4.1-4.14, 4.17, 4.30-4.31, 4.39-4.46, 4.61, 4.72, 4.86-4.90, 4.114- 4.120, 4.124 -4.131, 4.139, 4.114, 4.149, 4.156-4.161, 4.181-4.184, 5.5-5.29, 5.49-5.58, supplementary reference-3 and 4)

2. Analysis of soil [12 L]

a) Sampling of soil, sample preparation, Pre-treatment of Samples and Contamination, Trace Element Analysis, Sub-sampling, Drying Techniques, Milling, Grinding and homogenization,

b) Weighing and Dispensing: Weighing Errors, Dispensing Errors,

c) Acid-digestion, Ashing and Extraction Procedure: Acid-digestion and Washing: Aciddigestion of soils, Total soil nitrogen; Microwave acid-digestion, Dry ashing, Nitrate and water-soluble carbohydrate; Extraction Procedures for soils: pH extractants, Phosphate extractants, Potassium extractants, Trace element extractants,

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, Discussion - Determination of available nitrogen, Method-a: Determination of nitrate by selective ion electrode, Discussion - Determination of total mineralized nitrogen, Method-b: Determination of extractable ammonium-N, Method-b: Determination of extractable nitrate-N, Discussion, Determination of organic plus ammonium nitrogen, Method-a: Determination of soil nitrogen by auto analysis, Method-a: Reduction of nitrate before digestion and colorimetric auto analysis, Method-b: Determination of organic plus ammonium-N by digestion and distillation, Discussion, Determination of soil organic matter, Method-a: Determination of soil organic matter by loss on ignition, Method-b: Determination of easily oxidizable organic C by Tinsley's wet combustion, Discussion 5.8. Determination of pH and lime requirement, Method-a: Measurement of pH, Method-b: Determination of lime requirement, Method-c: Determination of pH in soils with soluble salts, Discussion - Determination of extractable phosphorus, Method-a: Determination of extractable phosphorus (manual method), Method-b: Determination of extractable phosphorus (automated method), Method-c: Determination of resin extractable phosphorus (automated method), Determination of extractable magnesium, potassium and Sodium, Determination of extractable trace elements, Discussion-Determination of extractable sulphur, Method-a. Determination of extractable sulphur (manual method), Method-b. Determination of extractable sulphur (automated method

(Ref-5: 17-35, 50-104, Ref.-6: 1- 14, 71-331)

References:

1. Standard methods for the examination of water and waste water, 23rd Ed. Rodger Baird, Andrew Eatson, Eugene Rice, jointly published by: American Public Health Association, American Water Works Association, Water Environment Federation.

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 Francis, 2018.

4. Environmental Chemistry, A. K. Day, New Age Publication Company

5. Methods in Agricultural Chemical Analysis: A Practical Handbook, N.T. Faithfull, CABI Publishing, Typeset by Wyvern 21 Ltd, Bristol (2002).

6. Soil Sampling and Methods of Analysis, Edited by M.R. Carter E.G. Gregorich, Canadian Society of Soil Science, Second Edition (2008)

Course Outcomes - At the end of course students should able to

CO-1: Define / understand various terms used in- analysis of water and soil

CO-2: Explain / describe techniques / methods of water and soil analysis

CO-3: Explain importance of water and soil analysis.

CO-4: Describe sources of water pollution and pollutants.

CO-5: Describe / explain methods / techniques of sampling of water and soil and their analysis.

CO-6: Solve numerical problems on analysis water and soil.

CHA606 (B) MJ: Automation and Sensor in Analytical Chemistry

[Optional Theory Paper, Two Credits] [30 L]

1. Introduction to laboratory Automation [2 L]

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. *(Ref-1: Relevant pages)*

2. Laboratory Automation [4 L]

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. *(Ref-1: Relevant pages)*

3. Flow Injection Analysis [6 L]

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. *(Ref-1: Relevant pages)*

4. Miniaturized Analytical systems [4 L]

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.*(Ref-1: Relevant pages)*

5. Chemical Sensors [4L]

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). *(Ref-1: Relevant pages)*

6. Biosensors in analysis [4L]

Introduction, producing biological surface, methods of immobilization, Achievement of biotransduction (amperometric, potentiometric, optical). *(Ref-1: Relevant pages)*

References-1: Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley –VCH

Course Outcomes - At the end of course students should able to

CO-1: Define / understand various terms used sensors and automation.

CO-2: Explain / describe techniques / methods in sensors and automation

CO-3: Explain importance sensors and automation in analytical chemistry.

CO-4: Describe application of automation in analytical laboratory and sensors.

CO-5: Give the choice of sensor for particular analysis.

CO-6: Explain principles of different types of sensors in analytical chemistry.

CHA-606(C) MJ: Forensic Analytical Chemistry

[Optional Theory Paper, Two Credits] [30 L]

1. Forensic Language and Definitions (3 L)

Defining Drugs, Origin of Drugs (Narcotics), Natural Drugs, Synthetic Drugs, Psychotropic Drugs (Mind Altering), Dependence and Addiction, Physical Dependence, Psychological Dependence, Drug Abuse, Hazards of Drug Abuse, Structural Relationships, Analogs, Designer Drugs, Isomers, Controlled Substance Statutes, Controlled Substances Act, Controlled Substances Laws, Schedule I to V, Controlled Substance: Charges and Offenses, Controlled Substance Submission to Crime Laboratories, Drug Cases in Crime Laboratories, Examination of Controlled Substances, Usable Quantity, Court Testimony, (Ref-1: 61-70)

2. Chemical Screening (3 L)

Introduction, Chemistry of Color Formation, Limitations of Chemical Color Tests, Chemical Color-Test Methods, Documentation, Chemical Color 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, Summary of Chemical Color Tests. (Ref-1: 78-90)

3. Microcrystal Techniques (3 L)

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, Gold Chloride Test, Gold Chloride in Phosphoric Acid Test, Platinum Chloride Test, Mercuric Iodide Test, Mercuric Chloride Test, Potassium Permanganate Test, Sodium Acetate Test, Critical Considerations, (Ref-1: 91-97

4. Alcohol (3 L)

Effects of alcohol on driving; Identifying the drug- or alcohol-impaired Driver, Tests of impairment, Alcohol measurement: *Blood*, *Breath*, Breath-alcohol instrumentation, *Urine and saliva* Urine, Oral fluid; Interpretation and presentation of alcohol results (**Ref-3**: 304-318)

5. Phenethylamines (6 L)

Introduction, Methyl Derivatives, Amphetamine: Introduction and History, Physical and Psychological Effects; Methamphetamine: Introduction and History, Physical and Psychological Effects; Phentermine: Introduction and History, Physical and Psychological Effects, Side Effects; Hydroxyl Derivatives, Phenylpropanolamine: Introduction and History, Physical and Psychological Effects; phedrine/Pseudoephedrine: Introduction and History, Physical and Psychological Effects; Ephedra Plant: Introduction and History; Ketone Derivatives: Cathinone, Methcathinone, Khat; Methylenedioxy Derivatives: 3,4-Methylenedioxyamphetamine, 3,4-Methylenedioxymethamphetamine; Methoxy Derivatives: Mescaline, Analytical Methods: Visual Inspection, Chemical Screening, Microcrystal Tests, Extraction Techniques, Extraction of Mescaline from Peyote; Confi rmatory Examination: Gas-Chromatography Mass Spectrometry, Fourier Transform Infrared Spectroscopy (**Ref-1**: 157-178)

6. Cannabis (4 L)

Introduction, History, Packaging for Forensic Examination, Forms of Cannabis, Psychoactive Ingredient, Forensic Identification of Marijuana, Botanical Identification, Macroscopic Properties, Microscopic Identification, Chemical Identification (Duquenois–Levine Test), Proposed Reaction Mechanism, Test Reagents, Test Technique, Thin-Layer Chromatography, Reagents, Test Technique, Visualization, Interpretation of TLC Results, Gas Chromatography Mass Spectrometry, Documentation, **(Ref-1**: 145-156)

8. Tertiary Amines: (2 L)

Introduction, Natural Tertiary Amines: Cocaine, Opiates: Morphine, Codeine, Heroin, Poppy; Synthetic Tertiary Amines: Phenylcyclohexylpiperidine; Analytical Methods: Visual Inspections, Chemical Screening of Tertiary Amines, Confirmatory Examination: Fourier Transform Infrared Spectroscopy, Gas-Chromatography Mass Spectrometry (**Ref-1**: 179-190)

9. Fire investigation (4 L)

Fire Investigation, Fire Debris Analysis, Preconcentration Methods, Data Analysis and Interpretation, Chemical Pattern Evidence, Detection Limits, Matrix and Substance, Weathering and Environmental Degradation, Forensic Investigation of Fire Deaths, Mechanism of Toxicity, Analytical Methods (Ref-431-463).

10. Forensic Analysis of Explosives (2 L)

Stand-Off Detection, Vapor Phase Detection, Spectroscopy, Laboratory Analysis of Explosives, Ion Chromatography, Mass Spectrometry, Integrated Example, (Ref-2: 488 – 507)

Reference-1: Basic Principles of Forensic Science**,** JaVed I. Khan, Thomas J. Kennedy, Donnell R. Christian, Jr. Humana Press, 2012.

Referenc-2: Forensic Chemistry, Suzanne Bell, Third Ed. Taylor and Francis

Reference-3: Clarke's Analytical Forensic Toxicology, Edited by Adam Negrusz, Gail AA Cooper, Pharmaceutical Press, UK, 2013

Course Outcomes - At the end of course students should able to

CO-1: Define / understand various terms used in- Forensic analysis

CO-2: Explain / describe techniques / methods of forensic analysis

CO-3: Explain importance of forensic analysis.

CO-4: Apply methods of forensic for spot investigation of Alcohols Fire and Explosive analysis.

CO-5: Describe / explain methods / techniques of forensic sampling and their analysis.

CO-6: Solve numerical problems on analysis forensic.

CHA-607(A) Practical: Measuring Water and Soil Quality

[Optional Practical Paper, Two Credits] [60 L]

Part-I: Water analysis (any six practical's)

1. Analysis of waste water /natural water sample for pH, dissolved oxygen, total dissolved salts (Conductometry) (Ref-1)
2. Analysis of waste water sample: turbidity, colour, total hardness (Ref-1 and 2)
3. Alkalinity and Buffering capacity of water (Ref-1)
4. COD of waste water sample (Ref-3) (Note: small scale experiment is possible where visible spectrometric method can be used for determination of Cr(VI) (Ref.-2)
5. Aqueous carbonate equilibria and corrosiveness (calcium carbonate saturation) (Ref-1, 2)
6. Biological oxygen demand (Ref-2)
7. Qualitative test for phosphate in soil sample and its estimation by colorimetry. (Ref-2, 3)
8. Pre-treatment to sulphide containing water (municipal waste water sample or artificially prepared water containing sulphide) its analysis for sulphide (Ref-2)
9. 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)

Reference:

1. Environmental Chemistry, Microscale Laboratory Experiments, Jorge G. Ibanez, Margarita Hernandez-Esparza, Carmen Doria-Serrano, Arturo Fregoso-Infante, Mono Mohan Singh, published by Springer.
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. Vogel's Textbook Quantitative Chemical Analysis, 6th Ed.

Part-II: Soil analysis (any six experiments)

10. Table work/field work (compulsory): Sampling and Sample Preparation, Measurement, Extraction, and Storage (Ref-1, Supplementary Ref-2 and 3)
11. Gypsum requirement of soil (Ref-1, Supplementary ref-2 and 3)
12. Determination of pH and lime requirement (Ref-1, Supplementary ref-2 and 3)

13. Micronutrient content in soil by AAS any two (Mn, Fe, Cu, Zn, Mo) (Ref-1, Supplementary ref-2 and 3)
14. Na and K content in soil by flame photometry (Ref-1, Supplementary ref-2 and 3)
15. Moisture content by LOD and dextermination of soil organic matter by loss on ignition or by wet oxidation method. (Ref-1, Supplementary ref-2 and 3)
16. Organic and ammonium nitrogen by Kjedahl's Method. (Ref-1, Supplementary ref-2 and 3)
17. Determination of nitrate by selective ion electrode. (Ref-1, Supplementary ref-2 and 3)
18. Determination of effective Cation exchange capacity (ECEC). (Ref-1, Supplementary ref-2 and 3)
19. Determination of easily oxidizable organic C by Tinsley's wet Combustion. (Ref-1, Supplementary ref-2 and 3)
20. Determination of extractable sulphur (manual method). (Ref-1, Supplementary ref-2 and 3)

References:

Ref-1: Methods In Agricultural Chemical Analysis A Practical Handbook, N.T. Faithfull, CABI *Publishing*

Ref-2: Handbook of Soil Analysis Mineralogical, Organic and Inorganic Methods Marc Pansu Jacques Gautheyrou, 2003 by Springer-Verlag , Berlin Heidelberg New York.

Ref-3: Soil Analysis Handbook of Reference Methods, Jr. Jones (Editor), CRC Press (2000)

Course Outcomes - At the end of course students should able to

CO-1: Define / understand various terms used in- analysis of water and soil

CO-2: Explain / describe techniques / methods of water and soil analysis

CO-3: Explain importance of water and soil analysis.

CO-4: Describe sources of water pollution and pollutants.

CO-5: Describe / explain methods / techniques of sampling of water and soil and their analysis.

CO-6: Able to perform calculations on analysis water and soil.

CHA-607(B): Practical Forensic Chemistry

[Optional Practical Paper, Two Credits] [60 L]

Note: For these practical aqueous solutions of the substance to be analysed at a conc. higher than detection limit should be given to the students.

Part-I: Any two

1. **Table work:** a) Collection, storage and transport of specimens b) Analytical toxicology worksheet c) Physical examination of samples. (Refence-1)
2. **TLC identification of drugs and poisons:** from list of compounds mixture of two to three compounds should be given for TLC identification. (Refence-1)
3. **Qualitative and Confirmatory Tests for poisons** (any ***four compounds*** from the least given in book): Test for aniline / para aminophenol, Test for antimony (No C.T.), Test for Borate (use talcum powder), Chlorate, Dinitrophenol pesticides, Ethanol / methanol, Formaldehyde, peroxides, Hypochlorites, Iodates, Nitrate / nitrite, Nitrobenzene, Oxalates;

Paracetamol, Phenol, Salicylic acid its derivatives, Thiocynates (**Note:** Sample in the form of aqueous solutions shall be given containing slightly higher conc. of poison than prescribed conc. in monograph of the substance). (Ref-1). For the substance tested student should write toxicity effects.

Part-II: Any six

Note: Sample in the form of aqueous solutions shall be given containing slightly higher conc. of poison than prescribed conc. in monograph of the substance.

5. Quantitative assay of borate (Ref-1)

6. Quantitative assay of Bromide (Ref-1)

7. Quantitative assay of ethanol (Ref-1)

8. Qualitative assay of Iron (III) (Ref-1)

9. Quantitative assay of Isoniazid (Ref-1)

10. Quantitative assay of Nitrite. (Ref-1)

12. TLC of organochlorine pesicides. (Ref-1)

13. TLC of organophosphorous pesticides. (Ref-1)

14. Quantitative assay of Paracetamol (Ref-1)

15. Quantitative assay of salicylates. (Ref-1)

16. Quantitative assay of Thiocynate (Ref-1)

17. Quantitative assay of toluene (Ref-1)

Part-III: Any four

18. Forensic analysis of finger print, tyre marks and foot ware impressions **(**Reference-2, 3)

(https://www.forensicsciencesimplified.org/prints/how.html), The Fingerprint Sourcebook; US department of Justice, **National Institute of Justice** *www.nij.gov*

19. **Forensic analysis of pen ink by TLC** (Reference-4)

(https://epgp.inflibnet.ac.in/epgpdata/uploads/epgp_content/S000016FS/P000695/M011494/ET/1516193607FSC_P8_M10_e-text.pdf)
(https://www.coursesidekick.com/chemistry/1203890);

(https://www.azolifesciences.com/article/Utilizing-Thin-Layer-Chromatography-in-Ink-Analysis.aspx)

(https://www.dvusd.org/cms/lib011/AZ01901092/Centricity/Domain/2935/Chromatography%20lab.pdf)

20. **Forensic analysis of lipstick by TLC / instrumental methods (**International Journal of Chemical and Molecular Engineering; Vol:13, No:5, 2019**); (**DOI: 10.1021/acs.jchemed.6b00942 J. Chem. Educ. 2017, 94, 1111−1117); (Forensic Science International, 17 (1981) 235 – 251)

21. **FTIR Analysis of cloth fibres** (**table work)** (DOI: 10.1021/acs.jchemed.6b00942 J. Chem. Educ. 2017, 94, 1111−1117); (*Molecules* 2022, *27*(13), 4281; https://doi.org/10.3390/molecules27134281);

(https://assets.thermofisher.com/TFS-Assets/CAD/Application-Notes/AN51517-E-ForensicCrime1013M-H-0115.pdf)

Note: FTIR spectra of different fabric materials such as cotton, woollen, nylon, polyester will be provided to the students. Students will draw chemical structure of polymer and will assign the peaks in FTIR to functional groups).

22. **Forensic Examination of hairs (table work)**

(https://www.asteetrace.org/static/images/pdf/01%20Forensic%20Human%20Hair%20Examination%20Guidelines.pdf): (https://www.azom.com/article.aspx?ArticleID=5528);

23. **Table Work:** GCMS and IR spectra analysis of some drug substances. (Referenc-4)

24. Forensic analysis a) hand writing, b) tyre marks and c) foot ware impressions.

25. Visit to Forensic laboratory.

Reference-1: Basics of Analytical Toxicology, World Health Organization, Geneva, 1995.

Reference-2: Basic Principles of Forensic Science**,** JaVed I. Khan, Thomas J. Kennedy, Donnell R. Christian, Jr. Humana Press, 2012.

Reference-2: Forensic Science_ An Introduction to Scientific and Investigative Techniques (4th Edition)-CRC Press (2014)

Referance-3: Forensic Chemistry Handbook, Edited by Lawrence Kobilinsky, Wiley, 2012

Referenc-4: Basic Principles of Forensic Chemistry; JaVed I. Khan, Thomas J. Kennedy, Donnell R. Christian, Jr. Humana Press,

Course Outcomes - At the end of course students should able to

CO-1: Define various terms used in forensic analysis.

CO-2: Explain / describe techniques / methods of forensic analysis

CO-3: Explain importance of forensic analysis in crime investigation.

CO-4: Describe importance of forensic analysis in investigation of drug and poison cases.

CO-5: Describe / explain methods / techniques of forensic sampling and their analysis.

CO-6: Perform calculations on forensic quantitative analysis.

CHA-608: Research Project

[Compulsory Paper, Four Credits] [Equivalent to 120 h]

GUIDELINE TO CARRY OUT PROJECTWORK

1. **Duration of Project work: -** One semester, 120 Laboratory hours. Each week 2 laboratory sessions of 4 hours should be allotted to each students.

2. College should allot research guide (mentor) to each student. ***Each student will be allotted separate project.***

3. **Choice of Research Problem and Workout:** Student should select research-based project with the help of his mentor. Research problem should be related to any branch of chemistry but preferably to any branch of analytical chemistry. Outline should be prepared by student with the help of mentor to perform and complete research project within stipulated time.

4. Internal Evaluation and Schedule for Submission of Project Work:

a. Experiment work must be completed by a student within 12 weeks from the commencement of the IIIrd semester.

b. Internal evaluation will be performed by mentor and one internal examiner when project is near to completion (10th week of semester).

c. Hard copy of the project work (two copies) should be submitted to department at the end of semester (15th week after commencement of IIIrd semester).

Format for submission of project -

The hard copy of project should contain about 30-40 pages (*A_4 size paper, 1 inch margin from all sides, font - Times New Roman, Font size – 12 pt*). Should be divided into the following parts:

a. Tittle page
b. Certificate of completion of Project Work from mentor and HOD.
c. Declaration by candidate regarding plagiarism
d. Index
e. **Chapter-1:** Introduction to problem (introduction, signification of research problems selected, aims and objectives) **(3 to 5 pages)**
f. **Chapter-2:** Review of Literature (Related Research Problem) **(8-10 pages)**
g. **Chapter-3:** Material and Methods **(6-8 pages)**
h. **Chapter-4:** Results and Discussion **(12 – 15 pages)**
f. **Chapter-5:** Conclusions **(1-2 pages)**
g. Bibliography
h. Acknowledgement

GUIDELINE FOR SUBMISSION AND ASSESMENT OF PROJECT WORK

1. Internal assessment 30% marks and external assessment 70% marks of 100 marks.

2. At the end of III semester two hard copies of research project must be prepared and submitted for certification and get both copies certified.

2. The certified copy of research project should be produced at the time of university project Examination by the candidate.

3. **External evaluation of project** – Power point presentation (20 minutes) by candidate followed by viva- voce exam purely based in project work.

Marks will be assigned to i) Project work report (design of problem and experiments, experimental work, accuracy in interpretation of results, discussions on results) – 35 marks; ii) power point presentation and explanations given on results – 20 marks, iii) question-answers – 15 marks.

4. After university project examination i.e. external evaluation of research project one copy must be submitted to department and one must be retained by the candidate.

Paper-wise Syllabus, Semester -IV

CHA-651 MJ: Applied Analytical Spectroscopy

[Compulsory Theory Paper, Four Credits] [60 L]

Section-I: Atomic absorption and emission Spectroscopy

1. Methodology for Trace Elemental Analysis [3 L]

Introduction, Analytical Terms and their Definitions, Units, Calibration Strategies, Presentation of Data: Tables, Presentation of Data: Graphs, Calculations: Dilution Factors, Quality Assurance and the Use of Certified Reference Materials. *(Ref-1: 1-13)*

2. Sample preparation techniques [5 L]

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 *(Ref-1: 17-36, Supplementary reference - 2)*

3. Sample Introduction system [3 L]

Introduction, Nebulizers, Spray Chambers and De-solvation Systems, Discrete Sample Introduction, Continuous Sample Introduction, Hydride and Cold Vapour Techniques. (*Ref-1: 39-56, Supplementary reference - 2)*

3. Atomic Absorption and emission Spectroscopy [7 L]

Introduction, Atomic spectra, Instrumentation of AAS: Automizers: Flame atomizer - premix burner, fuel gases and oxidants, graphite furnace, Hollow cathode lamps, spectrophotometers, detectors, Interferences in AAS (spectral and chemical), Quantitative analysis (calibration curve method, standard addition method, internal standard addition method), Practical applications of AAS from *(**Ref-4**: Relevant pages)*

3. Inductively Coupled Plasma AES and MS [10 L]

a. The Inductively Coupled Plasma and Other Sources: Introduction, inductively coupled plasma, Direct current plasma, microwave induced plasma, glow discharge.

b. Inductively Coupled Plasma AES: Fundamentals of Spectroscopy, Origins of Atomic Spectra, Spectral Line Intensity, Spectral Line Broadening, plasma spectroscopy, spectrometers, Detectors, charge transfer devices, interferences.

c. Inductively Coupled Plasma MS: Fundamental of MS, Inorganic mass spectroscopy, Interface, mass spectrometer, quadrupole mass analyser, sector field mass spectrometers, Ion-Trap Mass Spectrometer, Time-of-Flight Mass Spectrometer, detectors, interferences: Isobaric Interferences, Molecular Interferences, Polyatomic interferences, Doubly charged polyatomic interferences, Remedies for Molecular Interferences, Non-Spectral Interferences: Matrix-Induced, isotope dilution analysis, mass spectral interpretation. (Ref-1:57-117, supplementary *Ref- 2)*

4. Applications: [2 L]

Methods of Quantitation, Quantitative Analysis, Semiquantitative Analysis, Isotope Dilution, Internal Standardization, (Ref-2-123-131); Forensic analysis of documents, Clinical analysis of blood and urine, ***(Ref-1:** Relevant pages).* Analysis of metals from waste water sample of ICP-MS method *(Ref-3, section-3120, 3125)*

Reference

1. Practical Inductively Coupled Plasma spectroscopy, John R. Dean, Wiley India Pvt. Ltd. (AnTs Series book)

2. Practical Guide to ICP-MS, **Robert Thomas,** Third Ed. CRC Press, Taylor & Francis Group.

3. 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.

4. Vogels Quantitative Chemical Analysis, 6th Ed.

Section-II: Molecular Spectroscopic Methods

1. Molecular Absorbance and Derivative Spectroscopy [5 L]

Recapitulation of basic terms (transmittance, absorbance, molar absorptivity), Beets, law, limitation of Beer's law, deviation from Beer's law, Single and double instruments, Absorbance spectra, Quantitative analysis by absorbance measurement (scope, procedural details, derivative and dual wavelength spectroscopy) Photometric and spectrophotometric titrations. Ref-1: 332 – 394 Relevant part only).

2. Molecular Luminescence spectrometry [7 L]

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: *Components of Fluorometers and Spectrofluorometers*, Instrument Designs, Correction and Compensation Schemes, *Instrument standardization;* Applications of Photoluminescence Methods: *Methods for Organic and Biochemical Species, Phosphorometric method, Fluorescence Detection in Liquid Chromatography, Lifetime measurement, Fluorescence imaging;* **Chemiluminescence:** The Chemiluminescence phenomenon, measurement of chemiluminescence, analytical applications, problems. ***(Ref.-1:****395-425)*

3. Electron Paramagnetic Resonance Spectroscopy [10 L]

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, **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), (***Ref-3:****11-21, 27-49)*

4. Electron Spectroscopy for Surface Analysis [8 L]

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.* ***(Ref-2:*** *221-250, supplementary Ref-1).*

References:

1. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.

2. Materials Characterization, introduction to microscopic and spectroscopic techniques, Yang Leng, 2nd Wiley-VCH.

3. Introduction to Magnetic Resonance of Spectroscopy ESR, NMR, NQR, D.N. Sathyanarayana, I. K. International Publishing House Pvt. Ltd.

Course Outcome - At the end of course students should able to-

CO-1. Define / understand various terms in atomic absorption, atomic emission, fluorescence, ESR and electron spectroscopy.

CO-2. Explain instrumentation of atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.

CO-3. To describe basic principles of atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.

CO-4. Select appropriate methods for sample treatment in AAS / AES, ICPAES, ICPAES-MS.

CO-5. Explain advantages of ICPAES-MS over AES spectroscopy, fluorescence spectroscopy.

CO-6. Solve numerical problems on analysis all these spectroscopic methods.

CO-7. Interpret ESR spectra, super hyperfine splitting and g value in ESR, and parameters affecting it.

CO-8. Solve problems based on atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.

CHA-652 MJ: Chemical Methods of Pharmaceutical Quality Control

[Compulsory Theory Paper, Four Credits] [60 L]

Section-I: Pharmaceutical Dosage forms and general methods of quality control

1. Pharmaceutical Dosage Forms [8 L]

Pharmaceutical Dosage Forms: 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.(**Ref-2**: 13 - 47), Shelf life of pharmaceutical preparation.

2. Chemical Methods [12 L]

a. Identification: Identification test of organic substances/functional groups; Inorganic substances/ions (**Note:** Reactions taking place must be explained **see ref-3**); Identification of Barbiturates; Identification of Phenothiazines; Related Substances in Barbiturates; Related Substances in Phenothiazines; Related Foreign Steroids; Related Substances in Sulphonamides (**Ref-1:** 67 – 73 Supplementary **Ref-3:** 9 – 90)

b. Limit Test and Assay

Important Note: Write the chemical reaction and explain theoretical basis of the limit tests **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 (Ref-1: 74-80, Ref.- 4, 93-149)

Acetyl Value, Acid Value, Cineole, Ester, Ester Value, Hydroxyl Value, Iodine value, Methoxyl, Peroxide Value, Saponification Value, Assay of Steroids, Unsaponifiable Matter, Sulphur

Dioxide**,** Assay of Vitamin A, Assay of Vitamin D, Water-(Titration method and azeotropic distillation method), Zinc, Ethanol, Assay of Insulins (**Ref-1**: 80-99, Supplementary **Ref-3**: 104 to 150)

3. Pharmaceutical Methods of Determination [10 L]

Loss on drying and loss on ignition, melting range, 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. (**Ref-1:** 134-138; 175-188) Optical Rotation and Specific Optical Rotation Ref-1:138-139). Refractive index measurement (**Ref-1:** 163), Viscosity measurement (**Ref-1:** 163-165), Total Organic Carbon in water (**Ref-1:** 166).

References

1) Indian Pharmacopeia Volume I, 7th Ed

2) Indian Pharmacopeia Volume II, 7th Ed

3) Pharmaceutical Chemical Analysis: Methods for Identification and Limit Tests, Ole Pedersen, CRC press. Taylor & Francis Group, 2006.

Section-II: Analysis of Raw Materials and Active Ingredients

1. Quality of Analytical Data and Validation [6 L]

Instrumental Signals, Calibration Methods, External Standard, One Point Calibration, Internal Standard, Standard Addition, Normalization, Analytical Procedures, Validation, Specificity, Accuracy, Precision, Detection Limit, Quantitation Limit, Linearity and Range, Robustness, Test Methods in Ph. Eur. and USP, System Suitability, Adjustment of Chromatographic Conditions, Problems (**Ref-1**: 281 – 301).

2. **Chemical Analysis of Pharmaceutical Ingredients [12 L]**

Pharmaceutical Ingredients, Production, and Control, Pharmacopoeia Monographs, Melting point capillary method, (monograph on paracetamol and acepromazine malate tablet, acetaminophen, acetaminophen capsules, castor oil virgin, cefaclor), Impurities in Pharmaceutical Ingredients: Impurities in Pure Chemical Ingredients, Impurities in Organic Multi-Chemical Ingredients; Identification of Pharmaceutical Ingredients: IR Spectrophotometry (identification of ibuprofen, Identification of spironolactone**)** , UV-Vis Spectrophotometry (Identification of mianserin hydrochloride), Thin-Layer Chromatography (Identification of metrifonate**)**, Melting Point, Optical Rotation (Optical rotation for simvastatin**)**, Liquid Chromatography (Identification of calcitriol), Chloride (Identification of chloride in chlorcyclizine hydrochloride) and Sulfate, Identification, Impurity Testing of Pharmaceutical Ingredients (Pure Chemical Ingredients): Appearance of Solution (Appearance of solution for ibuprofen**)**, Absorbance (Absorbance and color of solution of esomeprazole magnesium**)** pH and Acidity or Alkalinity (pH of esmolol hydrochloride, Acidity or alkalinity of dopamine hydrochloride**)**, Related Substances (Related substances according to Ph. Eur. for omeprazole**)**, Residual Solvents (Limit of acetone in olmesartan medoxomil**)**, Elemental Impurities (Test for foreign zinc in human insulin**)**, Loss on Drying (Loss on drying for paracetamol**)**, Water (Determination of water in ephedrine), Assay of Pharmaceutical Ingredients, Aqueous Acid–Base Titration (Assay of omeprazole, amitriptyline hydrochloride, ephedrine hydrochloride, ephedrine), Non-Aqueous Acid–Base Titration (metronidazole benzoate, lidocaine**)**, Redox Titrations (ferrous fumarate), Liquid Chromatography (Assay of simvastatin), UV-Vis Spectrophotometry (Assay of hydrocortisone). (**Ref-3:** 305-369; 375 -388)

3. Chemical Analysis of Pharmaceutical Preparations [12 L]

Chemical Analysis of Pharmaceutical Preparations, Monographs and Chemical Analysis (BP monograph for paracetamol tablets**)**, Identification of the API: Identification by IR Spectrophotometry (Identification of aspirin, fluoxetine in fluoxetine hydrochloride oral solution, Identification of mupirocin in mupirocin calcium nasal ointment), Identification by Liquid Chromatography (Identification of fluoxetine in fluoxetine hydrochloride, droperidol in droperidol injection, Beclomethasone Dipropionate in Beclomethasone Dipropionate Ointment), Identification by UV-Vis Spectrophotometry (Identification of Diazepam in Diazepam Tablets, Flupentixol Decanoate in Flupentixol Decanoate Injection, Miconazole in Miconazole Nitrate Cream), Assay of the Active Pharmaceutical Ingredient, Assays Based on Liquid Chromatography (Assay of Omeprazole, Fentanyl in Fentanyl Citrate Injection, Assay of Hydrocortisone in Hydrocortisone Ointment), Assays Based on UV Spectrophotometry (Assay of Paracetamol in Paracetamol Tablets, Assay of Doxapram in Doxapram Hydrochloride Injection), Assays Based on Titration (Assay of Fe^{2+} in Ferrous Fumarate Tablets, Diphenhydramine in Diphenhydramine Hydrochloride Oral Solution), Chemical Tests for Pharmaceutical Preparations, Test for Related Substances (Related Substances in Paracetamol Tablets), Uniformity of Content (Uniformity of Content for Phenindione Tablets), Dissolution. **(Ref-3:** 391-332)

References:

Ref-1. Introduction to Pharmaceutical Analytical Chemistry, Stig Pedersen-Bjergaard, Bente Gammelgaard, Trine Grønhaug Halvorsen, Second Edition, Wiley (2012).

Ref-2. Pharmaceutical Drug Analysis, Ashutosh Kar, New Age International Pvt Ltd Publishers (2005).

Course Outcome - At the end of course students should able to-

CO-1. Define / understand various terms related to pharmaceutical identification, quality tests and assay.

CO-2. Explain methodology for identification, quality tests and assay of pharmaceutical raw materials and finished products.

CO-3. To describe basic principles of assay of raw materials and finished products.

CO-4. Select appropriate methods for analysis of raw materials and finished products.

CO-5. Explain importance of chemical analysis in quality control of pharmaceuticals.

CO-6. Solve numerical problems on analysis chemical analysis of pharmaceuticals.

CO-7. Interpret IR, UV-Visible, GC-Chromatogram and HPLC chromatogram in pharmaceutical identification.

CO-8. Apply physical methods for the quality control of pharmaceuticals.

CHA-653 MJ: Bio-Analytical Techniques

[Compulsory Theory Paper, two Credits] [30 L]

1. Introduction to Electrophoresis [6L]

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 ***(Ref-1:*** 1-70)

2. Capillary Electrophoresis: Basics, Instrumentation and Application [10 L]

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. ***(Ref-2:*** *5-33)*

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. ***(Ref-2:*** *103-141, 151-158)*

c) Qualitative and Quantitative Analysis and Applications: General Aspects of Qualitative and Quantitative Analysis, Application: Drugs and Natural Products, Amino Acids, Peptides and Protein ***(Ref-2:*** *243-246, 261-274, 278-303).*

2. Immunological methods of analysis [8 L]

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, Choice of Assay, **Stages in ELISA:** Solid phase (Immobilization of antigen on solid phase coating, coating time and temperature, 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-4, 5)**

Ref-1. Electrophoresis, Analytical chemistry through open learning Series, Wiley
Ref-2. Capillary Electrophoresis: Principles and Practice, R. Kuhn S. Hoffstetter-Kuhn, SpringerLaboratory, Springer-Verlag
Ref-3. Vogels's Textbook of Quantitative Chemical Analysis, 6th Ed.

Ref-4. Methods in Molecular Biology, Vol-42, ELISA-Theory and Practice, by John R. Crowther, Humana Press, Totowa, New Jersey.

Ref-5. Enzyme-linked Immunosorbent Assay (ELISA) From A to Z, Samira Hosseini, Patricia Vázquez-Villegas, Marco Rito-Palomares, Sergio O. Martinez-Chapa, published by Springer.

Course Outcome - At the end of course students should able to-

CO-1. Define various terms in electrophoresis, capillary electrophoresis, ELISA.

CO-2. Explain instrumentation paper electrophoresis, gel electrophoresis, capillary electrophoresis, and different types of ELISA.

CO-3. To describe basic principles paper electrophoresis, gel electrophoresis, capillary electrophoresis, different types of ELISA.

CO-4. To interpret experimentally obtained results of paper electrophoresis, gel electrophoresis, capillary electrophoresis, and different types of ELISA.

CO-5. Explain advantages and applications of paper electrophoresis, gel electrophoresis, capillary electrophoresis, and different types of ELISA.

CO-6. Apply particular method of analysis to particular type of sample.

CHA-654 MJP: Pharmaceutical Analysis for Quality Control

[Compulsory Practical Paper, Two Credits] [60 L]

Compulsory- practical's: Analysis of aspirin as per Indian Pharmacopeia**.** (Ref-2; 127-128)

1. **Aspirin:** Identification (B and C test); Appearance of solution; Clarity of solution in alkali; Sulphated Ash.

2. **Aspirin:** Limit tests: a) Heavy metals, b) Chlorides, c) Sulphates, d) Readily carburizable substances, e) Salicylic acid.

3. **Aspirin:** Loss on drying and assay.

4 to 6. Analysis of magnesium hydroxide as per Indian Pharmacopeia. (Ref-2; pp 714)

4. **Magnesium hydroxide -** Tests: Identification; Appearance of solution; heavy metals, iron and chloride

5. **Magnesium hydroxide -** Tests: Sulphate; calcium; soluble substances; substances insoluble in acetic acid;

6. **Magnesium hydroxide -** Loss on drying and assay

Alternative practical to Analysis of magnesium hydroxide

4 to 6. Analysis of calcium phosphate as per Indian Pharmacopeia. (Ref-2; pp 227-228)

4. **Calcium Phosphate:** Tests: Identification, acid insoluble substances, Heavy metals, Barium.

5. **Calcium Phosphate:** Tests: Carbonate, chloride, nitrate and Monocalcium and tricalcium phosphates, Reducing substances

6. **Calcium Phosphate:** Loss on ignition and assay (calibration of acid solution must be performed)

7. Compulsory: Identification test for Paracetamol tablet and tablet dissolution test on Paracetamol according to Indian Pharmacopeia (Ref-3: 902-903)

8. Identification test for Paracetamol table (according to IP); Average wt. of 20 tablets and UV-absorbance based assay of plane Paracetamol table using specific absorbance (British Pharmacopeia). (Ref-4: 419-421).

9. Analysis of Ca-Gluconate or calcium carbonate tablet or any Ca-supplementary tablet with respect to identification test, average wt. of 20 tablet, and Ca(II) content per tablet as per Indian Pharmacopeia. Express result as Ca-gluconate content ± Standard deviation. (Perform standardization of Na_2EDTA) (Ref-2; pp 224-225)

10. **Compulsory:** Non-aqueous titration: Interpretation of IR spectra of caffeine and assay of caffeine by **non-aqueous titration** method according to IP (Ref-2 pp 215) [standardize perchloric acid with potassium hydrogen phthalate]. **Or**

10. Interpretation of IR spectra of and assay of nicotine amide by **non-aqueous titration** method according to IP (Ref-2 pp 824) [standardize perchloric acid with potassium hydrogen phthalate].

11. Identification test for dextrose i.e. glucose (anhydrous or monohydrate); determination of a) specific optical rotation and b) water by Karl Fischer Method. (Ref-1 93, 138 and 2; 397-398)

12. Determination of NaCl and dextrose content in DNS saline solution: **a)** Cl by potentiometric titration and **b)** Dextrose by polarimetry (Ref-3: pp-1084).

13. a) Determination of refractive index of four liquids as per IP. b) Viscosity of ethyl cellulose by Oswald viscometer (use viscometer which comply specification of IP).

14. Identification and Assay of nicotinamide from tablets or capsules (VU-spectroscopy) (Ref-2: 824 – 825).

15. Identification test for Fe(II) and determination Fe(II) content in iron supplementary tablets (redox titration) (Ref-2: 803 – 807; Ref-4: 424 to 425).

16. Optical rotation and assay of ascorbic acid from tablet or from vit-C pure material by iodimetric titration (vit-C) (ref-2: 124-126).

17. Tablet friability and disintegration test; capsule disintegration test (Ref-1)

18. Related substances in paracetamol or in paracetamol tablet by thin layer chromatography (prefer to use readymade silica coated plate with silica gel GF254) (Ref-3: 900-901).

19. Assay of paracetamol in paracetamol syrup by liquid chromatography (Ref-3: 901-902).

20. Related substances in paracetamol syrup or in paracetamol tablet by liquid chromatography (Ref-3: 900-901; Ref-4: 393-395).

Compulsory: Table Work: a) Theoretical discussion on theory of identification of organic compound by IR spectroscopy and interpretation of IR spectra of at least three pharmaceutical compounds (Ref-4: 396 to 401; Ref-1: 107-112).

b) Calibration UV-Visible spectrophotometer as per IP (Ref-1)

References:

Ref-1. Indian pharmacopeia Vol-1, 2007

Ref-2. Indian pharmacopeia Vol-2, 2007

Ref-3. Indian pharmacopeia Vol-3, 2007

Ref-4. Introduction to Pharmaceutical Analytical Chemistry, Stig Pedersen-Bjergaard, Bente Gammelgaard, Trine Grønhaug Halvorsen, Second Edition, Wiley (2012).

Ref-5. Pharmaceutical Drug Analysis-New Age International Pvt Ltd Publishers (2005).

Course Outcome - At the end of course students should able to-

CO-1. Define / understand various terms related to pharmaceutical identification, quality tests and assay.

CO-2. Explain methodology for identification, quality tests and assay of pharmaceutical raw materials and finished products.

CO-3. To describe basic principles of assay of raw materials and finished products.

CO-4. Select appropriate methods for analysis of raw materials and finished products.

CO-5. Explain importance of chemical analysis in quality control of pharmaceuticals.

CO-6. Solve numerical problems on analysis chemical analysis of pharmaceuticals.

CO-7. Interpret IR, UV-Visible spectrum in pharmaceutical identification.

CO-8. Apply physical methods for the quality control of pharmaceuticals.

CHA-655(A) MJ: Analytical Methods of Food Quality Control

[Optional Theory Paper, Two Credits] [30 L]

1. Sampling and Sample Preparation [2 L]

Introduction, Selection of Sampling Procedures, Sampling Procedures, Preparation of Samples, Grinding, Enzymatic Inactivation, *(**Ref-1:** 71-80)*

2. Moisture and Total solids Analysis [2 L]

Introduction, Importance of Moisture Assay, Moisture Content of Foods, Forms of Water in Foods, Sample Collection and Handling, Oven Drying Methods: *General Information, Removal of Moisture, Decomposition of Other Food, Constituents, Temperature Control, Types of Pans for Oven Drying Methods, Handling and Preparation of Pans, Control of Surface Crust Formation (Sand Pan Technique), Calculations*; Vacuum Oven, Microwave Analyzer, Infrared Drying, Rapid Moisture Analyzer Technology, Distillation Procedures, Chemical Method: Karl Fischer Titration. *(**Ref-1** 87-96).*

3. Ash Analysis [2 L]

Introduction: *Definitions, Importance of Ash in Food Analysis, Ash Contents in Foods*; Methods: *Sample Preparation, Plant Materials, Fat and Sugar Products, Dry Ashing, Principles andInstrumentation, Procedures, Special Applications, Wet Ashing, Principle, Materials, andApplications, Procedures, Microwave Ashing, Microwave Wet Ashing, Microwave Dry Ashing, Other Ash Measurements, Comparison of Methods*

4. **Analysis of Lipids [5 L]**

a) Definition, Classification, General Considerations, Solvent Extraction Methods: Sample preparation, Solvent selection, Sample Preparation, Solvent Selection, Continuous Solvent Extraction Method: Goldfish Method, Semicontiguous Solvent Extraction Method: Soxhlet Method, Discontinuous Solvent Extraction Methods, Total Fat by GC for Nutrition Labelling (AOAC Method 996.06), 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. **(Ref.-1:** 119-130) **b) Characterization of Lipids** (bulk such as oils): 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, Problem on quantitative methods. ***(Ref-1:** 241, 246-258, Supplimentory-2, 3, 4).*

5. Proteins [5 L]

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's 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. (**Ref**-135 – 142, Supplimentory-2, 3). **b. Protein Characterization Procedures:** Amino Acid Analysis,Protein Nutritional Quality: Introduction, Protein digestibility, Protein efficiency ratio, and net-protein ratio, Other Protein Nutritional Quality Tests, Assessment of Protein Functional Properties, Determination of net protein utilization, digestibility and biological value, Problem on quantitative methods ***(Ref-1:** 271 - 277, Supplimentory-2, 3, 4)*

6. Carbohydrates: [5 L]

Introduction, Mono- and Oligosaccharides: Extraction, Total Carbohydrate: Phenol-Sulfuric AcidMethod, 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, Degree of Gelatinization of Starch, Degree of Retrogradation of Starch, Non-starch Polysaccharides, Dietary Fibres: Major Components of Dietary Fibre, General Considerations, Methods. Problem on quantitative methods ***(Ref-1**: 149-169 Supplimentory-2, 3).*

7. Analysis of Vitamins [3 L]

Introduction; Definition and Importance; Importance of Analysis; Vitamin Units; Chemical Methods; Vitamin-A; Vitamin E (Tocopherols and Tocotrienols); Vitamin E; Vitamin C; 2,6-Dichloroindophenol Titrimetric Method (AOAC Method); Microfluorometric; Method (AOAC Method); Thiamin (Vitamin B1) in Foods, Thiochrome Fluorometric Procedure (AOAC Method); Riboflavin (Vitamin B-2) in Foods and Vitamin Preparations, Fluorometric Method (AOAC Method); Comparison of Methods. Problem on quantitative methods ***(Ref-1**: 181; 188-195; Supplimentory-2, 3)*

Reference:

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

Course Outcome - At the end of course students should able to-

CO-1. Define various terms food analytical chemistry and food analytical techniques.

CO-2. Explain instrumentation used for food analysis.

CO-3. Describe basic principles of various methods of food analysis.

CO-4. Select / apply appropriate methods for sample treatment for particular analysis of food.

CO-5. Explain / describe role of food analysis in quality monitoring or control of food.

CO-6. Solve numerical problems on quantitative analysis of food.

CHA-655(B) MJ: Clinical Analytical Chemistry

[Optional Theory Paper, Two Credits] [30 L]

1. Collection of Specimens [2 L]

Blood: Collection of Blood specimens, storage and preservation, Urine: Collection of Urine, physical characteristics of urea, preservation and storage, Faeces: Collection and preservation. **(Ref.-1,** Relevant pages; Ref-2**)**

2. Analysis of Blood and Urine [6 L]

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. **(Ref.-1,** Relevant pages; Ref-2**)**

3. Determination of vitamins in body fluid [6 L]

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 D_3 (cholecalciferol), Vitamin E (Tocopherols, Determination of serum tocopherol by spectrophotometry by dipyridyl method), Vitamin B_1 (thiamine determination by flurometry), Vitamin B_2 (riboflavin, Photofluorometric method), Vitamin B_6 (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; Ref-2**)**

4. Therapeutic Drug monitoring by LC-MS [10 L]

a) Definition of Therapeutic Drug Monitoring, Definition of Toxicology **(Ref-2)**

b) Quantification of Eight Cannabinoids Including Cannabidiol in Human Urine Via Liquid Chromatography Tandem Mass Spectrometry **(Ref-2)**

c) Analysis of Benzodiazepines for Drug-Facilitated Assaults and Abuse Settings (Urine)

d) Targeted Opioid Screening Assay for Pain Management Using High-Resolution Mass Spectrometry **(Ref-2)**

e) Therapeutic Drug Monitoring of Lacosamide by LC-MS/MS **(Ref-2)**

f. LC-MS/MS Method for the Quantification of the Leflunomide Metabolite, Teriflunomide, in Human Serum/Plasma **(Ref-2)**

g. Quantification of Methotrexate in Human Serum and Plasma by Liquid Chromatography Tandem Mass Spectrometry **(Ref-2)**

h) Simultaneous Determination of Tacrolimus and Cyclosporine A in Whole Blood by Ultrafast LC-MS/MS **(Ref-2)**

References:

Ref-1: Varley's Practical Clinical Biochemistry, Gowenlock A. H., 6th Edition, 2006, CBS Publishers, New Delhi.

Ref-2: LC-MS in Drug Analysis Methods and Protocols, Second Edition, Edited by Loralie J. Langman, Christine L.H. Snozek, Humana Press.

Ref-3: Basic Concepts in Clinical Biochemistry: A Practical Guide; Vijay Kumar, Kiran Dip Gill, Springer.

Course Outcome - At the end of course students should able to-

CO-1. Define various terms in body fluid analysis, vitamin analysis, therapeutic drug monitoring.

CO-2. Explain instrumentations in body fluid analysis, vitamin analysis, therapeutic drug monitoring.

CO-3. Explain / describe basic principles of in body fluid analysis methods such as LC-MS, Lowry method, GOD-POD methos, urease method, fluorometric methods, colorimetric methods, etc.

CO-4. Apply / select particular method / instrumental parameters for analysis of particular sample.

CO-5. To interpret results of analysis of clinical sample.

CO-6. Solve numerical problems on analytical methods for body fluid analysis.

CHA-655(C) MJ: Analytical Techniques of Polymers Characterization

[Optional Theory Paper, Two Credits] [30 L]

1. Introduction [4 L]

Thermoplastics, Thermosets, Elastomers, High performance of polymers, copolymers, Blends, Cosmetics, Additives Speciality Polymers- liquid crystalline polymers, Conducting polymers, Thermoplastic elastomers, Biomedical polymers, biodegradable polymers. [3 L] (Ref-1: 1-28)

2. **Identification [4 L]**

Introduction, Preliminary Identification Methods: Solubility, Density, Behaviour on Heating; Infrared Spectroscopy, Raman Spectroscopy, Nuclear Magnetic Resonance Spectroscopy, Ultraviolet-Visible Spectroscopy, Differential Scanning Calorimetry, Mass Spectrometry, Chromatography, Emission Spectroscopy. (Ref-1: 31-64, Supplimentory-2)

3. Molecular Weight [4 L]

Introduction, Molecular Weight Calculations, Viscometry, Chromatography, Ultracentrifugation, Osmometry, Light Scattering, End-Group Analysis, Turbidimetric

Titration. (Ref-1: 103-119, Supplimentory-2)

4. Structural Methodology [4 L]

Introduction, Isomerism, Chain Dimensions, Crystallinity, Orientation, Blends, Thermal Behaviour, Dilatometry, Infrared Spectroscopy, Raman Spectroscopy, Nuclear Magnetic Resonance Spectroscopy, Optical Microscopy, Transmission Electron Microscopy, X-Ray Diffraction, Neutron Scattering, (Note: Thermal Analysis and thermal degradation are excluded as explained in TGA) ; (Ref-1: 121-149, 161-170, Supplimentory-2)

5. Degradation [5 L]

Introduction, Oxidative degradation, thermal degradation, radiation degradation, combustion, dissolution, infrared spectroscopy, Raman spectroscopy, electron spin resonance spectroscopy. Thermal analysis: -Thermogravimetric analysis, Differential Scanning Calorimetry, thermal mechanical analysis. Pyrolysis gas chromatography.

(Ref-1: 191-208).

6. Mechanical Properties [4 L]

Introduction, Stress-Strain Behaviour, Viscous Flow, Viscoelasticity: Creep, Models, Stress Relaxation; Elasticity, Processing Methods, Tensile Testing, Flexural Testing, Tear-Strength Testing, Fatigue Testing Impact Testing, Hardness Testing, Viscometry, Dynamic Mechanical Analysis. (Ref-1: 209-233).

References

1. Polymer analysis, Barbara H. Stuart, Analytical Techniques in the Sciences (AnTS), John Wiley and Sons Ltd.

2. Analytical Methods for Polymer Characterization Rui Yang, CRC Press Taylor& Francis Group, 2018.

Course Outcomes: At the end of course, students should able to -

CO- 1. Define / understand various terms in polymer analysis.

CO-2. Explain / describe techniques / methods of polymer analysis.

CO-3. To describe basic principles techniques / methods polymer analysis.

CO-4. Explain importance of polymer analysis.

CO-5. Choose suitable method / techniques to characterize quality of polymer

CO-6. Describe / explain results of analysis polymer.

CHA-656(A) MJP: Practical: Methods of Food Quality Determination

[Optional Practical Paper, Two Credits] [60 L]

Part-I: Non-instrumental methods (any 6)

1. Determination of total ash and acid insoluble ash in tea green tea. (Reference-1)
2. 1. Determination of water-soluble ash and alkalinity of water-soluble ash in tea green tea. (Reference-1)
3. Caffeine content in tea: Extraction of Caffeine from tea, its purification by crystallization, weigh it. Characterize the caffeine by UV visible and IR spectroscopy.
4. Determination of a) saponification value and acid value of an edible oil sample. (Reference-2)
5. Determination of a) Iodine Value and b) Rancidity of an edible oil sample. (Reference-2)
6. Detection (qualitative tests) of Adulterants in Milk (tests for any six adulterants). (Reference-3)
7. Determination of moisture and fat content in dried milk products. (Reference-3)

8. Determination of Milk Protein in Casein/Caseinates (Kjeldahl's method) (Reference-3)
9. Isolation of casein from milk thereby gravimetric estimation of casein from milk. (Ref-7).
10. Method for qualitative test for Vitamin A in Vanaspati ghee: Antimony trichloride method and Determination of carotenoid content of raw palm oil. (Reference-2)
11. Determination of Vit-C in lemon or in Vit-C supplement by indophenol or iodimetric method (Ref- 5 to 8).
12. Determination of dietary fibres in food sample (Ref-6)

Part-II: Instrumental Methods (any-6)

13. Quantitative Estimation of Urea in Milk (Reference-3)
14. Gas Chromatography-FID Method of Alcohol Estimation. (Reference-4)
15. Determination of Ethyl Alcohol Content - Dichromate Oxidation Method. (Reference-4)
16. Determination of Lead by Atomic Absorption Spectrophotometric (AAS) Method (Reference-4)
17. Determination of Calcium by Atomic Absorption or flame emission Spectrophotometric (AAS) Method. (Reference-4)
18. Total carbohydrates in food sample by Anthrone or by phenol sulphuric acid method. (Ref- 5 to 8)
19. Reducing sugar in food sample by Nelson Somyogi or DNSA method (Ref- 5 to 8).
20. Determination phosphate in food sample (Ref-5).
21. Determination iron in food sample (Ref-5).
22. Determination of Riboflavin in food sample by photoflurimetry.
23. Determination of purity of Sugar (Sucrose) and Dextrose (d-glucose) by polarimetry (Ref-9).
24. Separation and identification of amino acids in food sample by two dimensional TLC. (Ref-6,7)

References

1. FSSAI Manual of Methods of Analysis for Beverages Tea, Coffee and Chicory.
2. FSSAI Manual of Methods of Analysis for Oil and Fats
3. FSSAI Manual of Methods of Analysis for Milk and Milk products.
4. FSSAI Manual Analysis Alcoholic Beverages
5. Food Analysis, Edited by S. Suzanne Nielsen, Fourth Edition, Springer
6. Biochemical Methods, By S Sadashivan, A. Manickam; Third Edition, New Age International Publishers
7. An introduction to practical biochemistry, Third Ed. David T plummer, TATA-McGraw-Hill, Ed.

Course Outcome - At the end of course students should able to-

CO-1. Define various terms food analytical chemistry and food analytical techniques.

CO-2. Explain instrumentation used for food analysis.

CO-3. Describe basic principles of various methods of food analysis.

CO-4. Apply appropriate methods for sample treatment for particular analysis of food.

CO-5. Explain / describe role of food analysis in quality monitoring or control of food.

CO-6. To perform quantitative calculations on food analyses performed in laboratory.

CHA-656(B) MJP: Practical Clinical Biochemistry

[Optional Practical Paper, Two Credits] [60 L]

Minimum 12 experiments are to be performed

1. One or more visit to medical / pathological laboratory should be organized where routine biochemical analysis of urine and blood is performed so that students will get familiar with urea and blood collection method, their proper preservation and storage. Students will also observe some of the tests such as determination blood hemoglobin, or other sch test which cannot be performed in college laboratory. Student should prepare systematic report of visit as well as on blood and urea collection method, their proper preservation and storage.

Note: For further practical aqueous solution should be given containing analyte at same concentration as in blood or urine.

a) *The typical composition of urine of normal person is 0.05% Ammonia, 0.18% sulfate, 0.12 %phosphate, 0.01% Mg, 0.015% calcium, 0.6% K, 0.1% Na, 0.1% creatinine, water soluble Vitamins, 2% urea.* (For salts add ammonium sulfate, Na_2HPO_4, KCl, $CaCl_2$)

b) *Typical blood plasma composition: 100 ml blood plasma contains approximately: glucose 80 to 140 mg (normal person), total proteins 5 g, Calcium 7 mg, phosphate 11 mg, Mg 1.5 mg, Na 320 mg, Cl^- 100 mg, vit-c 0.5 to 2 mg, thiamine 2.5 to 6 microgram, riboflavin 3 to 19 microgram. etc.*

2. Glucose by glucose oxidase peroxidase method (use kit available in market).
3. Urea by Diacetyl Monoxime Method (do not use Kit, ask students to prepare kit like reagents).
4. Creatinine by trinitrophenol method (do not use Kit, ask students to prepare kit like reagents).
5. Cholesterol by $FeCl_3$ and acetic acid method or enzymatic method or by using kit available in market.
6. Estimation of Serum Proteins by Lowry method or by bitrate method.
7. Serum Calcium Estimation by Permanganate Titration or Cresolphthalein complexone colorimetric Method or by AAS.
8. Determination of Serum Inorganic Phosphate by colorimetry.
9. Separation amino acids by paper chromatography
10. Protein Paper Electrophoresis or Serum protein agarose gel electrophoresis
11. Estimation of Vit-C by titration method
12. Urine Na and K level by flame photometry
13. Riboflavin or Thiamine in urine or blood sample by Photoflurimetry

    Ref-1: Practical Textbook of Biochemistry for Medical Students, DM Vasudevan, Subir Kumar Das, Jaypee Brothers Medical Publishers (P) Ltd.

14. ELISA: Determination of Vitamin-C in serum samples by ELISA method. (https://www.cloud-clone.com/products/CEA913Ge.html **OR** any other)

15. ELISA: Determination of cotinine in serum samples (ready to use kits are available in market). https://www.liverpool.ac.uk/~agmclen/Medpracs/practical_5/practical_5.pdf)

16. ELISA: Quantitative in vitro determination of Gentamicin in milk and tissues (ready to use kits are available in market).

https://www.abcam.com/ps/products/287/ab287805/documents/Gentamicin-ELISA-Kit-protocol-book-v1-ab287805%20(website).pdf **OR**

https://fnkprddata.blob.core.windows.net/domestic/data/datasheet/WLP/5111GEN.pdf

17. **ELISA:** Any other ELISA experiment that is developed in your laboratory which can be performed to demonstrate steps in ELISA.

(https://www.canyons.edu/_resources/documents/academics/biology/elisa/ELISA_ver_B.pdf **OR** Biochemistry and Molecular Biology Education Vol. 37, No. 4, pp. 243–248, 2009)

17. SDS-PAGE: Separation of Proteins by SDS-PAGE. (David Plummer **OR** https://www.iitg.ac.in/biotech/MTechLabProtocols/SDS%20PAGE.pdf **OR** https://webstor.srmist.edu.in/web_assets/srm_mainsite/files/files/6%20SDS%20PHAGE.pdf **OR** https://www.sigmaaldrich.com/IN/en/technical-documents/protocol/protein-biology/gel-electrophoresis/sds-page).

18. SDS-PAGE: Any other SDS-PAGE or PAGE experiment designed in your laboratory.

Ref-1: Practical Textbook of Biochemistry for Medical Students; DM Vasudevan, Subir Kumar Das, Jaypee Brothers Medical Publishers (P) Ltd.

Ref-2: Varley's Practical Clinical Biochemistry; Gowenlock A. H., 6th Edition, 2006, CBS Publishers, New Delhi.

Ref-3: Basic Concepts in Clinical Biochemistry: A Practical Guide; Vijay Kumar, Kiran Dip Gill, Springer.

Course Outcome - At the end of course students should able to-

CO-1. Define various terms in clinical analytical chemistry.

CO-2. Explain instrumentations used in clinical analytical chemistry.

CO-3. Explain / describe basic principles of in body fluid analysis methods such as Lowry method, GOD-POD methos, urease method, fluorometric methods, colorimetric methods, ELISA, etc.

CO-4. Apply / select particular method / instrumental parameters for analysis of particular sample.

CO-5. To interpret results of analysis of clinical sample.

CO-6. To perform quantitative calculations on analyses performed in laboratory.

CHA-657: Research Project

[Compulsory Paper, Six Credits] [Equivalent to 180 h]

GUIDELINE TO CARRY OUT PROJECTWORK

1. **Duration of Project work: -** One semester, 180 Laboratory hours. In each week 3 laboratory sessions of 4 hours should be allotted to the students.

2. College should allot research guide (mentor) to each student.

3. **Choice of Research Problem and Workout:** Student should select research-based project with the help of his mentor. Research problem should be related to any branch of chemistry but preferably to any branch of analytical chemistry. Outline should be prepared by student with the help of mentor to perform and complete research project within stipulated time.

4. Internal Evaluation and Schedule for Submission of Project Work:

a. Experiment work must be completed by within 12 weeks from the start of IV semester.

b. Internal evaluation will be performed by mentor and one internal examiner when project is near to completion.

c. The final copy of the project work (two Copies) should be submitted to department at the end of semester (15^{th} week after commencement of IV semester.

Format for submission of project -

The project containing about 45-60 pages (*A_4 size paper, 1 inch margin from all sides, font - Times New Roman, Font size – 12 pt*). Should be divided into the following parts: -

a. Tittle page
b. Certificate of completion of Project Work from mentor and HOD.
c. Declaration by candidate regarding plagiarism
d. Index
e. **Chapter-1:** Introduction to problem (introduction, signification of research problems selected, aims and objectives) (6-8 Pages)
f. **Chapter-2:** Review of Literature (Related Research Problem) (12-15 pages)
g. **Chapter-3:** Material and Methods (8-10 Pages)
h. **Chapter-4:** Results and Discussion (20-25 Pages)
f. **Chapter-5:** Conclusions (1-2 page)
g. Bibliography
h. Acknowledgement

GUIDELINE FOR SUBMISSION AND ASSESMENT OF PROJECT WORK

1. Internal assessment 30% marks of 150 marks and External assessment 70% marks of 150 marks.

2. At the end of IV semester two copies of research project must be submitted for certification and get both copies certified.

2. The certified copy of research project should be produced at the time of university project Examination by the candidate.

3. Project evaluation – Power point presentation (20 minutes) by candidate followed by Viva- voce Exam purely based in project work. Marks will be assigned to i) Project work report (experimental work and accuracy in interpretation of results, discussions on results) – 50 marks; power point presentation and explanations given on results – 30 marks, question-answers – 25 marks.

4. After university project examination i.e. external evaluation of research project one copy must be submitted to department and one must be retained by the candidate.

Pattern for External Evaluation

Theory: For the four credit papers (70 marks) (paper with two sections); question paper will be set section wise. Each section will be of 35 marks. For two credit paper will be of 35 marks. Question paper format will be as follows:

Pattern for 4 credit paper

Time – 3 h 70 Marks 4 Credit

Section-I

Question-1 is compulsory; Solve any two questions from questions 2 to 4

Question-1 **A] Solve any 4 of the following (Define, reasoning, short problem, etc) [8]**

i)
ii)
iii)
iv)
v)

Question-1 **B] Problem [3]**

Question-2 **A] Answer the following [6]**

i)
ii)

Question-2 **B] Answer the following (Describe type) [6]**

i)
ii)

or single long answer for 6 marks

Question-3 **A] Answer the following (Explain/reasoning type) [6]**

i)
ii)

Question-3 **B] Answer the following [6]**

i) Tricky explanation / reasoning
ii) Problem

Question-4 **A] Answer the following (Notes) [6]**

i)
ii)

Question-4 **B] Answer the following [6]**

i) Tricky explanation / reasoning
ii) Problem

Section-II

Question-5 is compulsory; Solve any two questions from questions 6 to 8

Pattern same as section-I

Pattern for 2 credit paper

Time – 2 h 35 Marks 4 Credit

Pattern of question paper: as per section-I of 4 Credit paper

External Practical Examination:

1) One experiment to be performed by a candidate for 35 marks; Time – 4 hours

a) 30 marks will be allotted for practical work

b) 5 marks for oral.

For each practical course separate examines will appointed by university practical exam coordinator.

2. Project Examination: Poroject will be jointly evaluated by examines appointed to the practical exam coordinator. Project evaluation should be done as per guideline given in syllabus.

3. Student should produce hard copy of project / practical journal at the time of examination.