CBCS Syllabus M. Sc.-II Analytical Chemistry

To be Implemented form Academic Year 2020-21

Paper No	Semester - III	Credit
CCTP-7 CHA-390	Electrochemical and Thermogravimetric Methods of chemical analysis	4
CCTP-8 CHA-391	Analytical Method Development and Extraction Techniques	4
CCTP-9 CHA-392	Advanced Chromatographic Methods of Chemical Analysis	4
CBOP-3 Theory CHA-393	CBOP-3, CHA-393: A) Bioanalytical Chemistry	4
	Or	
	CBOP-3, CHA-393: B) Analysis of Food and Controlled Substances	4
CCPP-3 CHA-394	Basics of Instrumental Methods of Chemical Analysis	4
	Semester-IV	
CCTP-10 CHA-490	Advanced Analytical Spectroscopic Techniques	4
CCTP-11 CHA-491	Chemical Methods of Pharmaceuticals Analysis	4
CBOP-4 Theory CHA-492	CBOP-4: A) Laboratory Automation and Environmental Analytical Chemistry	4
	Or	
	CBOP-4: B) Analytical Chemistry of agriculture, polymer and Detergents	4
CBOP-5 Practical CHA-493	CBOP-5: A) Optional Analytical Chemistry Practical	4
	Or	
	CBOP-5: B) Project	4
CCPP-4 CHA-494	Applied Analytical Chemistry Practical	4

Equivalence to Old Syllabus

Old Paper (2014 pattern)	New syllabus (2020)
Semester - III	
CHA-390	CCTP-7, CHA-390
CHA-380	CCTP-8, CHA-391
CHA-391	CCTP-11, CHA-491
CHA-392	CBOP-4, CHA-492 (A)
CHA-387-Practical	CCPP-4, Practical, CHA-394
Semester - IV	
CHA-490	CCTP-10, CHA-490
CHA-491	CBOP-4(B), CHA-492 (B)
CHA-492	CBOP-4, CHA-492(A)
CHA-481	CBOP-3(A), CHA-393(A)
CHA-487, Practical	CCPP-4, Practical, CHA-494
CHA-488, Practical / Project	CBOP-5, CHA-493: A) Practical / B) Project

Details of Syllabus: Semester and Paper Wise

Semester-III

CCTP-7, CHA-390: Electrochemical and Thermogravimetric Methods of Chemical Analysis

Section-I: Electroanalytical Techniques

1. Coulometry [6 L)

Current voltage relationship during an electrolysis, Operating cell an at fixed applied potential, constant current electrolysis, Electrolysis at constant working electrode potential, Coulometric methods of analysis, Faradays laws of electrolysis, Instrumentations-Constant current and constant voltage instruments, potentiation coulometry-Instrumentation and applications, coulometric titrations - apparatus and applications, problems. ***(Ref.-1****:696-712, Ref-2: relevant pages)*

2. Voltammetry and Polarographic Methods of Analysis. [18 L]

a) Polarography (linear scan polarography): Polarographic principles, Instrumentation (different types of microelectrode such as dropping mercury electrode, the static drop mercury electrode, rotating disc and ring disc electrode, cell for polarography, reference and counter electrode and circuit diagram), polarogram and polarographic currents, charging or capacitive current, role of supporting electrolyte, factors affecting on polarographic wave, Ilkovic Equation, advantages and disadvantages of DME, polarographic maxima and maxima suppresors, interference due to dissolved oxygen, Applications (qualitative analysis, quantitative analysis by calibration curve and standard addition methods), specific examples of analysis – analysis of Cu, Cd, Zn, Pb, etc. from tap water and alloys., problems. ***(Ref-1:*** *716-723, Ref-2, Supplementary Ref. 3 and 4)*

b) Hydrodynamic Voltametry: Hydrodynamic voltametry and applications of hydrodynamic voltametry (volatameric detectors in chromatography and flow injection analysis, Voltametric oxygen sensors, amperometric titration. ***(Ref-1:*** *723-735)*

c) Cyclic Voltametry: Principle of cyclic Voltammetry, cyclic voltamogram of $K_3[Fe(CN)_6]$ and parathion (*Fundamental studies*), determination of analytes using CV, criteria of reversibility of electrochemical reactions, quasi-reversible and irreversible processes (***Ref-1****:735-742 Ref-2: Relevant pages, Supplementary* ***Ref.-5****: 27-68)*

d) Pulse Polarography: different types of excitation signals in pulse polarography, Differential pulse polarography, square wave polarography, Stripping method. Voltammetry with ultra-microelectrode, Applications of these technique 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. (***Ref-1: 742-753*** *2, Supplementary Ref. 3 and 4)*

References

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

5. Cyclic Voltammetery, Simultaneous Analysis and Reaction Mechanism, David K Gosser, VCH, 1994.

Section-II: Thermal Methods of Analysis

1. Introduction to Thermal Methods [2 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 [5 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 [7 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 [4 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 [4-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 [2 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.

CCTP-8, CHA-391: Analytical Method Development and Extraction Techniques

Sec-I: Analytical Extraction Techniques

1. Assay Validation and Inter Laboratory Transfer [2-L]

Introduction, fundamental definitions, Essential principles of method transfer, method validation report, the interlaboratory qualification (ILQ) process. *(**Ref-1:** pp 3 to 14)*

2. Statistical Analysis and analytical Figure of Merit [14 L]

Introduction, Errors (gross errors, systematic errors, random errors), accuracy, validation parameters: Accuracy, precision, mean and standard deviation, calibration, (linear response functions (linear regression-errors in slope and the intercept, error in the estimate of concentration, standard additions), non-linear response functions and weighted regression analysis, internal standards), selectivity and specificity (chromatographic methods), limits of detections (spectrophotometric methods, chromatographic methods and related techniques, receptor binding assay), limit of quantification, sensitivity, ruggedness and robustness, analyte stability in the sample matrix, how to reduce systematic errors, mean and standard deviation, reliability of results, confidence interval, comparison of results, comparison of two means of two samples, experimental design. ***(Ref-1:****15 to 68, Ref-2, p145-197)*

3. Overview of World Wide Regulations (2 L), ***Ref-1:*** *75 to 98)*

4. Specific methods and Applications: Dissolution Studies [4 L]

Introduction, Dissolution test, Apparatus – USP type –I and II, Sampling and analytical instrumentation, Single point test Vs. Dissolution profile, Calibration, regulatory guidelines, analytical validation, linearity, accuracy, precision, specificity. ***(Ref-1:*** *169 to 182)*

5. Specific Examples [2 L]

Explain these method w.r.t. method development and validation of specified analyte from these research papers. (***Ref-****4 to 7****)***

References

1. Development and validiation of Analytical Methods, Progress Pharmaceutical and Biomedical Analysis, Vol-3, Edited by Chitofer M. Riley and Tomas W. Rosanske (Elvier).
2. Vogel's Textbook of quantitative Chemical Analysis, Sixth Ed., Mendham, Denney, Barnes, Thomas, Pub: Pearson Education.
3. Development and validation of a colorimetric method for the quantitative analysis of thioamide derivatives, R.B. Ali et al., Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 220 (2019) 117154.
4. HPLC Method Development and Validation for Formaldehyde in Enteric Coating of ard Gelatine Capsules, Journal of Liquid Chromatography, 18(13), 2683-2693 (1995).
5. Development and Validation of Stability Indicating Rp-HPLC Method for Analysis Of Lercanidipine In Bulk Drug And Microemulsion Formulation, Journal of Liquid Chromatography & Related Technologies, 36:143–154, 2013.
6. Development and validation of an LC-MS/MS method for simultaneous quantification of voriconazole and its main metabolite voriconazole N-oxide in human plasma and its clinical application, Journal of Liquid Chromatography & Related Technologies, 40:20, 1047-1053.
7. Development and validation of the spectrophotometric method of butaphosphan determination in veterinary preparations, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 233 (2020) 118171.

Section: Analytical Extraction Techniques

1. Pre and Post Extraction Consideration [1 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)*

2. 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* ***Ref.-1:*** *39-45)*

3. Solid Phase extraction (SPE) [6 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)*

4. Solid phase micro-extraction [6 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, stirbas sorptive extraction, liquid phase micro-extraction, , membrane micro extraction, micro extraction in packed syringe). ***(Ref-1:** 85-110, Ref-3)*

5. 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 Microextraction, A Practical Guide, Edited by Sue Ann Scheppers Wercinski, CRC press, Taylor and Francis.

CCTP-9, CHA-392: Advanced Chromatographic Methods of Analysis

Section-I: Mass spectrometry and Gas Chromatography

1. Mass Spectrometry [6 L]

Fundamentals, Electron ionization, Chemical ionization, Instrumentation: *Quadrupole mass spectrometers, Magnetic sector mass spectrometers, TOF mass analyser, detector;* Interpretation of mass spectra, Types of ions Isotopic abundances and characteristic ion clusters, Nitrogen rule and rings-plus-double-bonds, steps in interpretation, Examples ***(Ref-1:** 39-72, Supplementary Ref.- 4)*

2. Fundamentals of Chromatographic Methods of Analysis [4 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)*

3. Gas Chromatography [4 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)*

4. Gas Chromatography-Mass Spectrometry [8 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 [2 L]

Ref-4: 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- 5: 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)*

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 [4 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 [6 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 [2 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 [2 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 [8 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)*

Chapter-6: Super Critical Fluid Chromatography and Extraction [2 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)*

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.

CBOP-3, CHA-393: A) Bioanalytical Chemistry
B) Analysis of Food and Controlled Substances

CBOP-3, CHA-393: A) Bioanalytical Chemistry

Section-I: Bioanalytical Techniques

1. Introduction to Electrophoresis [6 L]

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).*

3. HPTLC and Detectors for HPTLC [8 L]

Thin layer chromatography, High performance thin layer chromatography. *(Ref-3)*

Planar Chromatography Detectors, Transmittance Measurements in Thin-Layer Chromatography, The Lambert-Beer Law, Reflectance Measurements in TLC and HPTLC, The Kubelka–Munk Equation, Reflectance Measurements with a Diode-Array Scanner, Spatial Resolution on the Plate, Spectral Distribution on HPTLC Plates, Spectral Evaluation Algorithm, Mass Spectrometric Detection in TLC, Direct Plate Extraction (SSSP), MALDI Techniques (MALDI-MS), Atmospheric Pressure Mass Spectrometry. Applications. ***(Ref-4:** 231-257)*

References:

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

Sec-II: Clinical Analytical Chemistry

1) Analysis of blood and urine [12 L]

a) Collection of Specimens: Blood: Collection of Blood specimens, storage and preservation, Urine: Collection of Urine, physical characteristics of urea, preservation and storage, Faeces: Collection and preservation. **b) Analysis of Blood and urine**: Determination of blood and plasma glucose by glucose oxidase method, Determination of urine for glucose, Determination of ketone bodies in blood, Oral Glucose tolerance test, Determination of serum creatinine, estimation of serum bilirubin, Estimation of serum cholesterol, determination of blood haemoglobin, Urate: determination of serum urate, Determination of urea in urine by urease method and by direct colorimetry, Estimation of Na, K, Ca by flame photometry, inorganic phosphate by colorimetry. **c) Determination of vitamins in body fluid:** Classification of vitamins with example, Each vitamin must be explained with respect of functions, deficiency diseases, daily requirement, and analytical method i) Retinol (determination of retinol and serum carotene in serum using TFA), Vit 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**)**

2. Immunological methods of analysis [10 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, Choise of Assay, **Stages in**

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

3. Radioimmunoassay [2 L]

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

References:

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

CBOP-3, CHA-393: B) Analytical Methods of Food and Controlled Substances

Section-I: Analytical methods of Food

1. Introduction to Food Analysis [1 L]

(Ref-1: 1-13)

2. Sampling and Sample Preparation [1 L]

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

3. Moisture and Total solids Analysis [1 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*; Distillation Procedures, Chemical Method: Karl Fischer Titration. ***(Ref-1** 87-96).*

4. Ash Analysis [1 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 and Instrumentation, Procedures, Special Applications, Wet Ashing, Principle, Materials, and Applications, Procedures, Microwave Ashing, Microwave Wet Ashing, Microwave Dry Ashing, Other Ash Measurements, Comparison of Methods*

5. **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).*

6. 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 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)*

7. Carbohydrates: [5 L]

Introduction, Mono- and Oligosaccharides: Extraction, Total Carbohydrate: Phenol-Sulfuric Acid Method, total reducing sugars by Nelson Somyogi method, Specific Analysis of Mono- and Oligosaccharides - High-performance Liquid, Gas Chromatography, Enzymic Methods, Chromatography, Mass Spectrometry, Thin-layer Chromatography, Polysaccharides: Starch, Total Starch, Degree of Gelatinization of Starch, Degree of Retrogradation of Starch, Non-starch Polysaccharides, Dietary Fibres: Major Components of Dietary Fibre, General Considerations, Methods. ***(Ref-1**: 149-169 Supplimentory-2, 3).*

8. Determination of food preservatives [5 L]

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

References

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

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

Section-II: Analytical Methods of Controlled Substances

1. The narcotic drug and Psychotropic Substances (NDPS) Act-1985 [1 L]

Important Definition: *Drug, Cannabis (Indian Hemp), Cannabis Products, Coca-derivatives, Coca Leaf, Coca Plant, Illicit Traffic, Controlled Substance, Manufactured Drug, Opium, Opium Poppy, Poppy Straw, Poppy Straw Concentrate, Psychotropic Substance,* Prohibition Control and Regulation of NDPS (**Ref.-1**: 122-134, Ref-2)

2. Chemical Screening and Microcrystal Tests [2 L]

a) Chemical tests: Introduction, Chemistry of Color Formation, Limitations of Chemical Color Tests, Chemical Color-Test Methods, Documentation, Chemical Colour Tests: *Chen's Test, Dille–Koppanyi's Test, Mecke's Test , Marquis' Test, Nitric Acid Test, Primary Amine Test, Secondary Amine Test, Tertiary Amine Test, Van-Urk's Test, Duquenois–Levine Test, Froehde's Test, Janovsky Test, Weber Test.* **b) Microcrystal Techniques**: Introduction, Advantages of Microcrystal Techniques, Disadvantages of Microcrystal Techniques, Documentation, Microcrystal Test Techniques, Aqueous Test Technique, Volatility Test Technique, Acid and Anionic Test Technique, Aqueous Test Reagents, **(Ref-3:** 79-95)

3. Analysis of Drugs/Narcotics [21 L]

a) Amphetamine and Related Compounds: Introduction, Qualitative Identification of Amphetamines, Sampling and Physical Description of Amphetamines, Presumptive Testing of Amphetamines, Thin Layer Chromatography of Amphetamines, Definitive Identification of Amphetamines, Quantification of Amphetamines, Comparison and Profiling of Amphetamine Samples, The Leuckart Synthesis of Amphetamine, The Reductive Amination of Benzyl Methyl Ketone, The Nitrostyrene Synthesis, Impurity Extraction and Sample Comparison. ***(Ref.-4:** 13-34)*

b) **The Analysis of LSD:** Introduction, Qualitative Identification of LSD, Sampling and Physical Description of LSD Blotter Acid, Extraction of LSD Prior to Analysis, Presumptive Testing for LSD, Thin Layer Chromatography of Samples Containing LSD, Confirmatory Tests for the Presence of LSD ***(Ref.-4:** 37-43)*

***c) Cannabis sativa* and Products:** Introduction, Origins, Sources and Manufacture of Cannabis, Analytical Sequence, Bulk and Trace Sampling Procedures, Qualitative Identification of Cannabis, Identification of Herbal Material, Identification of Other Materials, Comparison of Cannabis Samples. ***(Ref.-4:** 49-65)*

d) **Diamorphine and Heroin:** Introduction, Origins, Sources and Manufacture of Diamorphine, Appearance of Heroin and Associated Paraphernalia, Bulk and Trace Sampling Procedures, Identification, Quantification and Comparison of Heroin Samples,

Presumptive Tests for Heroin, Thin Layer Chromatography of Heroin Samples, Gas Chromatographic–Mass Spectroscopic Identification of Heroin, Quantification of Heroin Samples, Comparison of Heroin Samples ***(Ref.-4:** 73-92)*

e) **Cocaine:** Introduction, Origins, Sources and Manufacture of Cocaine, Extraction and Preparation of Coca Paste, Synthesis of Pure Cocaine, Qualitative Identification of Cocaine, Presumptive Tests for Cocaine, Thin Layer Chromatography, Definitive Identification of Cocaine, Quantification of Cocaine, Quantification of Cocaine by GC–MS, Quantification of Cocaine by UV Spectroscopy, Comparison of Cocaine Samples. ***(Ref.-4:** 97-109)*

f) Products from *Catha edulis* and *Lophophora williamsii*: Introduction, Products of Catha edulis, Identification, Quantification and Comparison of Khat Samples, Comparison of Khat Samples, Products of Lophophora williamsii, Physical Description and Sampling of Materials, Presumptive Tests for Mescaline, TLC Analysis of Mescaline, HPLC Analysis of Mescaline, GC–MS Analysis of Mescaline, Comparison of Peyote Samples. ***(Ref.-4:** 113-124)*

g) Analysis Barbiturates and Benzodiazepines: Introduction, Analysis of Barbiturates and Benzodiazepines, Extraction of Barbiturates and Benzodiazepines from Dose Forms, Presumptive Tests for Barbiturates and Benzodiazepines, TLC of Barbiturates and Benzodiazepines, Confirmatory Analysis of Barbiturates and Benzodiazepines, Quantification of Barbiturates and Benzodiazepines, Introduction, Products of *Catha edulis*, Identification, Quantification and Comparison, of Khat Samples, Comparison of Khat Samples, Products of *Lophophora williamsii*, Physical Description and Sampling of Materials, Presumptive Tests for Mescaline, TLC Analysis of Mescaline, HPLC Analysis of Mescaline, GC–MS Analysis of Mescaline, Comparison of Peyote Samples. ***(Ref.-4:** 139-149).*

Reference

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

CCPP-3: Basics of Instrumental Methods of Chemical Analysis

Section-1: Analytical method Development and Validation

Expt. No	**Name of Experiments**
	Compulsory experiment
1	**Demonstration Practical:** a. Calibration of UV-Visible spectrophotometer for control of absorbance as per IP or BP b) Theoretical basis for the choice of solvent for recording UV-Visible spectra of substances c) Theoretical basis for choice proper concentration for recording the UV-Visible spectrum d) Recording the UV-Visible spectrum of any one substance like caffeine, aspirin, paracetamol, $KMnO_4$ or any other substance of interest having characteristic UV-Visible absorbance i) identification of characteristics peaks in spectrum, b) Choice of λ_{max} for quantitative analysis c) Calculation of Molar

	absorptivity (ε) and d) Sp. absorbance (absorbance of sample solution for 1% solution). Theoretical interpretation of spectra. (Ref-6,7)
2	**Table Work:** a) Theorical basis of method development and validation – Accuracy, precision, noise level, detection limit, quantitation limit, Calibration curve and standard addition method and theoretical basis of choice between two, b) Expression of results: Calculation of mean, standard deviation, error and absolute error, elimination of data, c) Regression analysis of calibration curve and its importance. (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 or non-metal ion or organic substance with respect to: a) Selection of ligand / reagent and colour formation method b) Choice of reaction cond. such as concentration of analyte and colour forming reagent, pH for colour formation reaction, etc. c) Determination of λ_{max} for quantitative analysis d) estimation of noise level, detection limit, quantisation limit and linearity range (Calculate R^2 value). Thereby set conc. limits for calibration curve method and standard addition method. e) Estimation of known of metal ion by calibration curve method and by standard addition method in triplicate for the validation of method. f) Estimation of metal ion from sample by calibration curve method and by standard addition method in triplicate (Regression analysis must be performed for both methods and results shall be accepted when R^2 is greater than 0.95) g) Detection of possible interfering metal ion. Some suggested examples: i) Colorimetric / visible spectrophotometery Cu(II) or Co(II) – R-nitrososalt and estimation of Cu(II) (Sample - alloy brass or bronze or coin) (Cent. Eur. J. Chem. 10(5), 2012, 1617-1623, DOI: 10.2478/s11532-012-0081-7) ii) Colorimetry / visible spectrophotometery Mn(II)-Formaldoxime or Mn(II)-oxidized to $KMnO_4$ (sample - tea leaves on ashing or plant micronutrient supplement). (Ref.-1, 7) iii) Colorimetry / visible spectrophotometery B as borate with curcumin reagent (Sample – Talcum powder can be taken) (Ref-1 and 3) iv) Colorimetry / visible spectrophotometery Ni(II) or Co(II) by alfa nitroso beta-napthol (Sample - Steel alloy). Ref-1 and Monatshefte Fur Chemic 111, 1413 1425, Springer-Verlag 1980 **Note: A student can select any other metal and own synthesized ligand system under the guidance of his mentor.** **Some examples of non-metal ions** i. NO_3^- or NO_2^- by colorimtry / visible spectrophotometery (Ref- 1 and 2) ii. NH_4^+ or NH_3 by colorimetry / visible spectrophotometery (alkaline phenol-perchlorate reagent) (Ref-1 and 2) iii. SO_4^{2-} by Colorimetry / visible spectrophotometeryetric (Ref-1) **Examples of Organic substances** i) Analysis of aspirin Colorimetry (Ref. 11) ii) Assay of Vitamin-C by Colorimetry from lemon or orange juice (Ref- 12)

	iii) Colorimetry / visible spectrophotometery phenolic compounds (Salicylic acid, salbutamol sulfate, phenol) by Folin-Ciocalteau reagent (Ref-4) v) Colorimetry / visible spectrophotometery Analysis of paracetamol (Ref-8) **Note:** i) A mentor can practice multiple examples in batch**.** ii) *Student shall prepare systematic report in the form of journal which will 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.*
	Any three experiments
8	Analysis of Riboflavin by visible spectrometry and Photoflurometry. Compare results with respect to sample requirement, detection limit, accuracy of both methods. Give your choice for analysis of i) Riboflavin as raw material in pharmaceutical industry and ii) blood/ urine/vitamin supplement. Explain reason for choice of method. *(Ref-4, 6 and 9).*
9	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)*
10	Determine amount of $NaHCO_3$ from eating soda sample or from mixture of $NaHCO_3$ + Na_2CO_3: Determine amount of $NaHCO_3$ by thermal decomposition method (gravimetry) on burner as well as 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)*
11	Perform pH metric titration for estimation of CH_3COOH from vinegar using i) 0.1 M standardized NaOH simultaneously using phenolphthalein indicator and pH meter ii) 0.5 M standardized NaOH using pH meter. Compare the results of three methods and give your comment. *(Ref-3)*
12	Determine aspirin in tablet conventional titration and by potentiometric titration and compare the results of two method. *(Ref-10)*
13	Development of turbidimetric method for estimation of i) PO_4^{3-} at low level using ammonium molybadate reagent or ii) S^{2-} using reaction with suitable metal ion such as Cu(II). iii) estimation of Mg(II) by Nessler's reagent***. (for self development)***
14	**Qualitative and confirmatory test for (minimum four)** Test for aniline / para aminophenol, Test for antimony / mercury (No C.T.), Test for Borate (use talcum powder), Dinitrophenol pesticides, Ethanol / methanol, Formaldehyde, Hypochlorites, Iodates, Nitrate / nitrite, Paracetamol, Phenol, Salicylic

	acid its derivatives, Thiocynates (**Note:** Aq. Solutions shall be given containing prescribed conc. in monograph of the substance). (Ref-13).
	References: 1. Separation, Preconcentration and Spectrophotometry in Inorganic Analysis, by Z. Marczenko and M. Balcerzak, Analytical Spectroscopy Library – 10, Elsevier 2. Standard methods for the examination of water and wastewater, 23rd Ed. Roger B. Baird, Andrew D Eaton, Eugene W. Rice, American Public Health Association, Americal water works association, Water environment federation. 3. Vogels textbook of Inorganic Quantitative Analysis, 4. Biochemical Methods, Third Edition, By S Sadashivan, A. Manickam; New Age International Publishers 6. Indian Pharmacopeia: 2007, Vol-1, 2, 3. 7. Chemical Analysis and Material Characterization by spectrophotometry, Bhim Prasad Kafle, Elsevier 8. Ultraviolet and Visible Spectrophotometry in Pharmaceutical Analysis, Sandor Gorog, Published by CRC press, Taylor and Fransis. 9. An introduction to Practical Bichemistry, David T. Plummer, Tata McGraw-Hill publishing Company Ltd. 10. Experiments in chemistry, D. V. Jahagirdar, Himalaya Publishing Company 11. Method Development for Analysis of Aspirin Tablets, Journal of Chemical Education, Volume 65 Number 10 October 1988. 12. Vitamin C as a Model for a Novel and Approachable Experimental Framework for Investigating Spectrophotometry, Journal of Chemical Education, DOI:10.1021/acs.jchemed.9b00197. 13. Basic Analytical Toxicology, R. J. Flangan, R. A. Braithwait, S. S. Brrown, B. Viddop, F. A. de Wolff, published by WHO.
	Section – II: Introduction to Analytical Techniques (12 experiments)
	Nitrogen Estimation
1	Estimation organic nitrogen by Kjeldahl's Method or semi-micro Kjedahl's method (example: milk powder, soil sample, cooked food containing pulses, fertilizer etc.). *(Ref-1)*
	Solvent Extraction
2	Extraction of organic substance by Soxhelet or semi micro Soxhelet extraction (such as Essential oils, carotenoids from carrot, Caffeine for tea powder) and their isolation from solvent. Purity by TLC, BP/MP. *(Ref-7)*
3	Isolation of carotenoids from spinach / lycopene from tomato. TLC / column separation to find out number of carotenoids. *(Ref-6)*
	Solid Phase Extraction, Ion exchange chromatography
4	Determination of Ion exchange capacity of ion exchange resins *(Ref-1).*
5	**Solid Phase Extraction:** Isolation of amino acids from aqueous sample using ion exchange resin and their identification by colorimetric test (very dilute glycine solution can be used as an example of alfa amino acid) *(Ref. 5)*

	Or Isolation of caffeine using RP C-18 cartridge from cold drinks and quantitative estimation *(Ref-7)* Or Isolation of beta carotene from spinach leaves on silica gel cartridge by solid phase extraction and its quantification visible spectrophotometry. *(Ref-7)*
6	**Pre conc. 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
7	Flame photometric analysis of water /soil sample for Na^+ and K^+ by calibration curve method (give regression analysis for both curves) *(Ref-1)*
8	Estimation of K^+ from soil/water sample by internal standard and its confirmation by standard addition method (give regression analysis of both curves) *(Ref-1)*
	Methods of Trace Analysis of metals: Atomic Absorption Spectroscopy
9	**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)*
10	Estimation of any two-metal ion by atomic absorption spectroscopy from soil or micronutrient supplement or food sample. *(Ref-1)*
	Turbidimetry / Nephelometry
11	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)*
12	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
13	Estimation of quinine sulphate from tablet by calibration curve and its confirmation by standard addition method. *(Ref-1)*
14	Determination of Zn(II) by Photoflurimetry *(Ref-1)*
	Polarimetry
15	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 in DNS saline and glucose supplement (Glucon-D) sample by polarimeter. *(Ref-2)*
	Quantitative TLC

16	Separation of Colours by TLC / Paper chromatography, their isolation by elution from paper or TLC and quantification by colorimetry. *(Ref-1)*
17	Analysis of the Composition of a Mixture of Nitroanilines by Thin-Layer Chromatography and Ultraviolet/Visible Spectrometry *(Ref.-8)*
	HPLC
17	**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)*
18	Estimation of APC tablet by HPLC method *(Ref-1, 3, 8)* or HPLC method developed in your laboratory.
	Gas Chromatography
19	**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)*
20	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
21	**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.
22	TGA analysis of dolomite ore for $CaCO_3$ and $MgCO_3$ content *(Ref-1)*
23	TGA analysis $CuSO_4\,5H_2O$ *(Ref-1)*
	Cyclic Voltammetry
24	Cyclic voltmmetric study of Fe(II)/Fe(III) system. Basic principle and calculation of basic parameters from CV. *(Ref-1, 10, 11)*
25	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).
	Students Self activity
1	**a) Compulsory:** Prepare report on construction, working, representation, uses and care of electrodes: Calomel electrode, silver-silver chloride electrode, platinum electrode, conductivity cell, combine glass electrode. (***Ref-1).*** b) Actual construction of standard silver-silver chloride as reference electrode (Replacement to saturated calomel electrode as it contain highly toxic Hg(II), Hg(I) and Hg metal), salt bridge and their testing. ***(Ref-9).***

	c. Construct graphite electrode using graphite rod or used dry pen-cell. Perform redox titration using graphite electrode prepared by you and calomel as reference electrode. Perform same titration using Pt electrode and calomel electrode. Report do 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 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.

Semester-IV

CCTP- 10, CHA-490: Advanced Analytical Spectroscopic Techniques

Section-I: Atomic Spectroscopic Methods

1. Sample preparation techniques **[2 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)*

2. Atomic Absorption and emission Spectroscopy [6 L]

Introduction, Atomic spectra, Instrumentation of AAS: Sample introduction system: Nebulizers, Laser Ablation technique, hydride vapour generators, automizers: Flame atomizer - premix burner, fuel gases and oxidants, graphite furnace, hydride generator, cold vapour technique, 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-3. (**Ref-3**: Relevant pages, Supplementary references 4,5)*

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

a. Inductively Coupled Plasma AES: Introduction to Atomic emission spectroscopy, inductively coupled plasma, Direct current plasma, microwave induced plasma, glow discharge, plasma spectroscopy, spectrometers, Detectors, interferences.

b. Inductively Coupled Plasma MS: Fundamental of MS, Inorganic mass spectroscopy, Interface, mass spectrometer, quadrupole mass analyser, detectors, interferences, isotope dilution analysis, mass spectral interpretation. ***(Ref-1:**57-117, supplementary Ref- 6)*

c. Applications: 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-2, sec. 3120, 3125)*

4. Atomic Fluorescence Spectroscopy [6 L]

Atomic fluorescence, Apparatus for AFS, EMR source for AFS, LASERS, Cells for AFS, Plasmas- ICP and DCP, Detectors, theory of AFS, Analysis with AFS, Interferences with AFS, Resonant ionization Spectroscopy, LASER enhanced ionization spectroscopy. *(Ref-5)*

5. Elemental Analysis [2 L]

Particular analyses, Elemental organic microanalysis, Total nitrogen analysers (TN), Total sulphur analysers, Total carbon analysers, problems on empirical and molecular formula on CHONS analysis. ***(Ref. -7:** 441-450)*

Reference

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

Section-II: Molecular Spectroscopic Methods

1. Molecular Luminescence spectrometry [6 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 Design, 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, Flurescence imaging;* **Chemiluminescence:** The Chemiluminescence phenomenon, measurement of chemiluminescence, analytical applications, problems. ***(Ref.-1:****399-426)*

2. Electron Paramagnetic Resonance Spectroscopy [12 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, relaxation process, transition probability. **Hyperfine Structure:** Nuclear hyperfine splitting, radical containing one proton, spin Hamiltonian, selection rules, radical containing a set of equivalent protons, radical containing a set of multiple protons, radical containing multiple sets of protons (I = ½), radical containing multiple sets of proton (I > ½), Atomic radicals, Origin of hyperfine interaction, sigma radicals, assignments of spectra using Huckel MOs, alternant hydrocarbons, hyperfine splitting constants, second order splitting, Applications. *(**Ref-3:** Relevant pages, Supplementary Ref-4)*

3. Electron Spectroscopy for Surface Analysis [6 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).*

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.
4. Structural Methods in Molecular Inorganic Chemistry, DavidW. H. Rankin, Norbert W. Mitzel, Carole A. Morrison, Wiley (John Wiley & Sons, Ltd.), 2013

CCTP-11, CHA-491: Chemical Methods of Pharmaceuticals Analysis

Section-I: Pharmaceutical Dosage forms and General Methods Analysis

1. Pharmaceutical Dosage Forms [4 L]

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: 14 - 47),* Shelf life of pharmaceutical preparation.

2. Chemical Test, Limit Test and Assay [6 L]

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

a) Limit Tests: Aluminium, Aluminium in Adsorbed Vaccines, Arsenic, Calcium in Adsorbed Vaccines, Chlorides, Heavy metals, Iron, Lead, Potassium, Sulphates, Sulphated Ash, Total Ash, Free Formaldehyde, N-N-Dimethylaniline *(Ref-1: 74-80, Ref.- 4)*

b) Assays: Acetyl Value, Acid Value, Cineole, Ester, Ester Value, Hydroxyl Value, Iodine value, Nitrogen, Methoxyl, Nitrite Titration, Peroxide Value, Saponification Value, Assay of Steroids, Unsaponifiable Matter, Assay of Vitamin A, Assay of Vitamin D, Water-(*Titration method and azeotropic distillation method*), Zinc, Ethanol, Assay of Insulins *(Ref-1: 80-99, Ref-3)*

3. Pharmaceutical Methods of Determination [6 L]

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: 175-188*)

4. Microbiological Assay of Pharmaceuticals [8 L]

Biological assay in general, **a) Agar diffusion assay – Quantitative basis:** *Introduction, The theory of zone formation, what happens in practice, principles of calculation of potency estimate*; **b) The Theory and Practice of Tube Assay- Growth promoting substances**: *Introduction, the mode of action of growth limited by amino acids, growth limited by vitamins, production of acid by lactobacilli, clinical factor in the assay of growth promoting substances*; c) **The Theory and Practice of Tube Assay-Growth Inhibiting Substances:** *Introduction, measurement of response, the forms of response line, historical development of the turbidimetric method, linearization of sigmoid curve, the quantitative theory of microbial growth and inhibition, a practical determined log dose – response curve, factor affecting finals cell count, the influence of temperature, the influence of time,* **d) What do we want assay**: *pharmacopeial intension and control of antibiotic bulk materials, control in routine in manufacture, Research and development*; d**) General Practical Aspects of Microbiological Assay:** Introduction, test solutions (weighing – sample of unknown, dilution of primary solution to test level, problem with very dilute solutions, the assay medium), selection of Latin squares and plating routine, Aspects of technique (the test organism, inoculating the medium, assay plate, assay tube, diluents, the sample, test solution and the effect of contamination, application of test solution-agar diffusion assay, application of test solution-turbidimetric assay; Calculation of potency, **e) Standard and reference materials** *(Ref-4: 1, 9-18, 23-35, 37-56, 59-64, 65-77, 79-84, Ref-1: 45-52)*

Section-II: Analysis of Raw Materials and Active Ingredients

1. Introduction to Pharmaceutical Analytical Chemistry [1 L]

Introduction, Official European Pharmacopoeia definitions, Pharmaceutical Analytical Chemistry, Manufacture of Pharmaceuticals, Development of New Drugs, Use of Pharmaceuticals ***(Ref-3:** 1-7)*

2. Marketing Authorizations, Pharmaceutical Manufacturing, and International Pharmacopoeias [1 L]

Introduction, Marketing Authorization and Industrial Production, Pharmacopoeias, Life Time of Pharmaceutical Preparations and Ingredients. ***(Ref.3:** 9-14)*

3. 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, caster 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***)**, Foreign Anions (*Test for foreign chlorides and sulfates in furosemide*), Sulfated Ash (*Residue on ignition for acetaminophen***)**, Elemental Impurities (*Test for foreign zinc in human insulin***)**, Loss on Drying (*Loss on drying for paracetamol***)**, Water (*Determination of water in ephedrine*), Identification and Impurity Testing of Organic Multi-Chemical Ingredients: *Oxidizing Substances,* ***Only importance of the should be explained*** *- Acid Value, Hydroxyl Value, Iodine Value, Peroxide Value, Saponification Value, Unsaponifiable Matter),* Other Tests (*Chromatographic profile for peppermint oil***)**, 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:** 305-388)*

4. Chemical Analysis of Pharmaceutical Preparations [10 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

1) Indian Pharmacopeia Volume I, 7th Ed
2) Indian Pharmacopeia Volume II, 7th Ed
3) Introduction to Pharmaceutical Analytical Chemistry, Stig Pedersen-Bjergaard, Bente Gammelgaard, Trine Grønhaug Halvorsen, Second Edition, Wiley (2012).
4. Pharmaceutical Chemical Analysis: Methods for Identification and Limit Tests, Ole Pedersen, CRC press. Taylor & Francis Group, 2006.

CBOP-4, CHA-492: A) Laboratory Automation and Environmental Analytical Chemistry Or CBOP-4, CHA-492: B) Analytical Chemistry of agriculture, Polymer and Detergents

CBOP-4, CHA-492: A) Laboratory Automation and Environmental Analytical Chemistry

Sensor-I: Laboratory Automation and Sensor Based Techniques

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

Sec-II: Environmental Analytical Chemistry

1. Water Pollution and Measurement of Water Quality [16 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—Multi-Wavelength Method, Turbidity, Odour, Taste, Acidity, Alkalinity, Calcium Carbonate Saturation, (Introduction, Indices Indicating A Water's Tendency To Precipitate Or Dissolve Caco3, Indices Predicting The Quantity Of $CaCO_3$ 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). **Metal ions:** Introduction, Preliminary Treatment Of Samples *(Introduction, Filtration for Dissolved and Suspended Metals, Treatment for Acid-Extractable Metals, Digestion for Metals, Nitric Acid Digestion, Nitric Acid-Hydrochloric Acid Digestion, Nitric Acid-Sulfuric Acid Digestion, Nitric Acid-Perchloric Acid Digestion, Nitric Acid-Perchloric Acid Hydrofluoric Acid Digestion, Dry Ashing, Microwave-Assisted Digestion*), Quantitative analysis by AAS, FES and ICPAES: *Only general explanation as this part is covered in detail in Analytical spectroscopy* Sec-I. (**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). **c) Inorganic non-metal:** Introduction, Determination of Anions By Ion Chromatography, Inorganic Anions

By Capillary Ion Electrophoresis; Bromide (phenol red method), cyanide, Chlorine (DPD colorimetric method), Fluoride (ion selective method, complexone method), ammonia (titrimetric method, ions elective method and phenate method), NO_2^- - colorimetric method, NO_3^- (nitrate electrode and Cd reduction method), Organic nitrogen by Microkjeldahl method, Dissolved oxygen (iodometric and membrane electrode method), phosphate (molybadate – $SnCl_2$ - colorimetric method), Sulfide (methylene blue and ion selective method), **d) Organic constituents:** Biochemical oxygen demand, Chemical oxygen demand, total organic carbon, phenols (direct photometric method), surfactants. *(**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. Air Pollutants and Analysis of the Atmosphere and Air Pollutants [8 L]

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

b) Pollutant Analysis: Atmospheric Monitoring, Air Pollutants Measured, Sampling, Methods of Analysis, determination of Sulfur Dioxide, Nitrogen Oxides, Analysis of Oxidants, Contents, Analysis of Carbon Monoxide, Determination of Hydrocarbons and Organics, Determination of Specific Organics in the Atmosphere, Analysis of Particulate Matter**,** Filtration, Collection by Impactors, Particle Analysis, X-Ray Fluorescence, Determination of Lead in Particulate Matter, Direct Spectrophotometric Analysis of Gaseous Air Pollutants. *(**Ref-2:** 707-718).*

Reference

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

CBOP-4, CHA-492: B) Analytical Chemistry of agriculture, Polymer and Detergents

Section-I: Agricultural Analytical Chemistry

1. Analysis of soil [10 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: *Acid-digestion 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**-1: 17-35, 50-104, Ref.-2: 1-14, 71-331)*

2. Fertilizer Analysis: [6 L]

Discussion -Determination of total nitrogen in presence of nitrate and organic, Method-a: Determination of total nitrogen in presence of nitrate and organic N, with final determination by distillation, Method-b: Determination of total nitrogen in presence of nitrate and organic N, with final determination by auto-analysis, Discussion - Determination of phosphorus in fertilizers, Method-a. Determination of water-soluble phosphorus (extraction), Method-a: Determination of water-soluble phosphorus, (auto-analysis), Method-a: Determination of water-soluble phosphorus (manual method), Method-b. Determination of 2% citric acid-soluble phosphorus – method for basic slags (Thomas phosphate), Method-c: Determination of total phosphorus in the acid digest from Method-b. with final determination by auto-analysis, Discussion-Determination of potassium in fertilizers, Method-a: Determination of water-soluble potassium, Method-b. Determination of ammonium oxalate-soluble potassium, Method-c: Determination of potassium in the acid digest from, Liming Materials, Determination of the moisture and neutralizing value of liming materials, Determination of fineness of grinding. ***(Ref.-1:** 106-123)*

3. Analysis of Pesticide Residues [8 L]

Preparation of Samples, Collection and Preparation of Soil Samples, Collection and Preparation of Water Samples, **Individual Pesticide Residue Analytical Methods:** Aldicarb(GC), Captafol (GC Method), Captafol (HPLC), Captan (HPLC), Chlorothiophos (GC), Ehty;ene Thiourea (GC), Folpet (HPLC), 1,naphyl acetic acid (GC), Paraquat (photometric); **Multiple Pesticide Residue Analytical Methods:** Substituted Phenyl Urea Herbicides (GC), Organochlorine and Organophosphorus Pesticides (GC and TLC), Dithiocarbamate and Thiuram Disulphide Fungicides (potometric), Phthalimide fungicides (HPLC). *(**Ref-3:** 17-23, 87-116, 135-148, 167-172, 241-250, 297- 307, 353-359, 401-406).*

References:

1. Methods in Agricultural Chemical Analysis: A Practical Handbook, N.T. Faithfull, CABI Publishing, Typeset by Wyvern 21 Ltd, Bristol (2002).
2. Soil Sampling and Methods of Analysis, Edited by M.R. Carter E.G. Gregorich, Canadian Society of Soil Science, Second Edition (2008)
3. Manual of Pesticide Residue Analysis Volume I, Edited by Hans-Peter Thier and Hans Zeumer, Pesticides Commission, VCH, New York.

Sec-II: Analytical Chemistry of Polymer and Detergents

Section-II: Polymer Analysis and Detergent analysis

1. Polymer analysis

a. Introduction **1 L**

Introduction and Types of polymers. *(**Ref-1: 1-28)***

b. 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)*

c. Molecular Weight **3 L**

Introduction, Molecular Weight Calculations, Viscometry, Chromatography, Ultracentrifugation, Osmometry, Light Scattering, End-Group Analysis, Turrbidimetric Titration. *(**Ref-1:** 103-119, Supplimentory-2)*

d. 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)*

e. 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).*

2. Analysis of Surfactants **[8 L]**

a) Surfactant types; classification, identification, separation: Why analyse surfactants, Features peculiar to surfactant analysis, Basic Definitions (surfactant, anionic surfactant, cationic surfactant, non-ionic surfactant, amphoteric surfactant, weakly acidic and basic surfactants), Common types of surfactants of all four classes, b) **Elemental analysis:** Metals, Determination of nitrogen, Determination of sulphur, Determination of phosphorus. c) **Basic techniques:** Extraction of surfactants (Liquid-solid extraction: *Liquid-liquid extraction using separating funnels, Liquid-liquid extraction using extraction columns),* Acid-base titration**:** *(general principles, end point detection, Determination of weak acids and bases and their salts,* Potentiometric titration: *Principle, Applications, Methods for esters, amines, alcohols and unsaturated fatty materials, Two-phase titration of ionic surfactants with surfactants of opposite charge, Introduction, ISO 2271: Principle and procedure, Potentiometric titration with surfactants of opposite charge using a surfactant-sensitive electrode, Advantages of potentiometric titration, Construction and performance of surfactant-sensitive electrodes, Titration procedure,* Open-column chromatography. d**) Analysis of Representative surfactants: i) Analysis of Anionics:** Introduction, general methods- *Para-toluidine precipitation/titration method,* Analysis of Alkane sulphonates: *Determination of total alkane sulphonate, Determination of mean molecular weight of alkane monosulphonates, Separation and determination of alkane mono- and disulphonates,* Carboxylates: *Titration with benzethonium chloride, Solvent extraction, Acid-base titration, Determination of soaps in fatty products,* **ii) Analysis of nonionics:** Analysis of Ethoxylated alcohols, alkylphenols and fatty acids: *Composition, Determination by potentiometric titration, Determination by the cobaltothiocyanate colorimetric method, Determination of total nonionics and polyethylene glycols, Volumetric determination of polyethylene glycols, Determination of oxyethylene groups, Fatty acid ethoxylates: determination of polyethylene glycols, free fatty acid and mono- and diester;* **iii) Analysis of cationics and amphoterics:** Introduction, Analysis of Quaternary ammonium salts:, *Two-phase titration with sodium dodecyl sulphate, Two-phase titration with sodium tetraphenylborate, Determination of free amine and amine hydrochloride,* Amines: *Determination of molecular weight and total, primary, secondary and tertiary amines*
(Ref-3: 1, 8, 17-24, 31-36, 42-75, 105-109, 119-124, 142-143, 149-160, 171-177, 222-226, 264-280, 310-317, Supplementary reference-4)

Reference

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
3. Introduction to Surfactant Analysis, Edited by D. C. Cullum, Springer-Science + Business Media, B.V, 1994.
4. Handbook of Detergents, Editor-In-Chief Uri Zoller, Part-C, Heinrich Waldhoff, Rüdiger Spilker, Marcel Dekker, New York, 2005.

CBOP-5, CHA-493: A) Optional Analytical Chemistry Practical or
CBOP-5, CHA-493: B) Project

CBOP-5, CHA-493: A) Optional Analytical Chemistry Practical

	Section-I: Any 12 experiments
2	**Table Work:** Characterization of organic compounds by VU-Visible, IR and NMR spectroscopy (any two compounds, Example- paracetamol and aspirin - actual spectra must be given for analysis)
	Analytical Chemistry for Self-Employment: Preparation / Isolations Analytical Standards or reference material for analytical laboratories **(Imp. Note:** all these materials can be used for further experiments)
3-4	Solvent extraction: Isolation and purification caffeine. Impurity present if any by TLC. Indian Pharmacopeia Tests: identification tests, MP, loss on drying, Total heavy metal and assay. (Spectral characterization may be performed) (Ref-5)
5-6	Synthesis of Paracetamol (or any other medicinal compound) by green chemistry route and recrystallization. Test as per IP: TLC, MP, Identification tests, limit test for chloride, LOD and assay. (spectral characterization may be performed) (Ref-5 and 4)
7-8	Synthesis, recrystallization of ligands used in analytical chemistry: Example- diethyldithiocarbamate (or other dithiocarbamate ligand), salicylaldehyde ethylenediamine, 8-Hydroxyquinoline, or any other, purity by suitable method. (Packaging and labelling - student self-work).
9	Preparation of Analytical Reagent Kit (any one) which will contain all the reagents for determination of specific analyte, labelling and packaging of reagents and writing of standard protocol to use the kit and testing of kit, detection limits should be mentioned. (such kits are used in commercial analytical laboratories) (You cannot adopt procedures from commercialized kits which available in market). Suggested examples: a student can select other example with permission of his mentor. 1) Analysis of creatinine (trinitrophenol method) (Ref-3) 2) Blood cholesterol (ferric chloride method) (Ref-3) 3) Analysis of proteins by Lowry method (Ref-3, 6) 4) Analysis of reducing sugar by colorimetry method. (Ref-3, 6) 5) Regents for qualitative test of carbohydrates and protein for higher secondary laboratories – Fehling solution-A, Fehling solution-B, Iodine solution, Molisch reagent, Barford reagen, Benedicts reagent, Seliwanoff reagent, Bials reagent, biuret reagent. (Ref-6) 5. Preparation of standard solutions required for limit tests of pharmaceuticals as per I. P. **(Note:** These kits should be used for experiments and can be prepared 1 week before the schedule of such an experiment)
10	Synthesis of Methyl red indicator, purification, MP/ TLC and test for colour change with respect to change in pH of indicator, (packaging and forwarding – student self-work) (Ref-8)
11	Identification of amino acids / sugars / or any other mixture by two-dimensional chromatographic method (TLC or paper) (Ref.-3)

12	Separation of Chloride and Bromide on anion exchange resin (Ref.-1)
13	Sephadex gel separation of proteins (Ref-6)
14	Determination of molecular weight by gel permission column chromatography (Sephacryl S-200 column) (Ref-6)
15	Separation of leaf pigments by adsorption Chromatography (Ref-6)
16	Separation of amino acids by ion exchange chromatography (Ref-6)
17	Separation of proteins by ion exchange (DEAE cellulose) chromatography (Ref-6)
18	Analysis of phenolics in Aurvedic solution / syrup preparations (Ref-3)
19	Apply Limit test of heavy metals and iron to Aurvedic medicinal preparations (Ref-4)
	Section – II: 12 experiments
	Part-I: Volumetric and Gravimetric methods for quantitative analysis of complex materials (Not more 6)
1-2	Analysis of Cement SiO_2, Calcium, Iron, Magnesium and Aluminium (Ref-1)
3-4	Analysis of mixed fertilizer sample for total nitrogen, K and phosphate content. (Ref-1)
5	Analysis of dolomite ore with respect to SiO_2, Ca and Mg (Ref-1)
6	Analysis of brass alloy for Cu and Sn (Ref-1)
7	Determination of total Ash, Ash Insoluble in Hydrochloric acid, Alkalinity of soluble ash in coffee [FSSAI manual]
8	Determination of total sugars or Glucose content in glucose supplement by (glucon-D) by titration with Fehling solution (FSSAI manual)
9	Electrogravimetry determination of Cu(II) or Ni(II) (Ref-1)
	Part-II: Instrumental Methods of selective analysis from complex materials or mixtures (Not more than-4)
10	Analysis of fertilizer Micronutrient Supplement for Fe, Mn, Cu, and B. **Colorimetry**: Fe with thiocyanate, Mn as $KMnO_4$, B using curcumin reagent, and Cu using diethyldithiocarbamate ligand. (Ref-1, 2)
11	Estimation of thiamine by photoflurimetry from multivitamin capsule by calibration curve and its confirmation by standard addition method. (Ref-3)
12	Analysis of Chloride, Bromide and Iodide from mixture by potentiometry (Ref-1)
13	Use of ion selective electrodes for determination (F, Cl, Ca, NH_4^+ etc. from water)
14	TGA/DTA analysis of polymer for binders, polymer content, etc. (Ref-7)
15	Determination of Ca in milk powder by flame photometry by standard addition or calibration curve method (FSSAI Manual]
	Part-III: Chromatography
16	Identification of amino acids / sugars / or any other mixture by two-dimensional chromatographic method (TLC or paper) (Ref.-3)
17	Separation of Chloride and Bromide on anion exchange resin (Ref.-1)
18	Sephadex gel separation of proteins (Ref-6)

19	Determination of molecular weight by gel permission column chromatography (Sephacryl S-200 column) (Ref-6)
20	Separation of leaf pigments by adsorption Chromatography (Ref-6)
21	Separation of amino acids by ion exchange chromatography (Ref-6)
22	Separation of proteins by ion exchange (DEAE cellulose) chromatography (Ref-6)
23	**Students self-activity - Compulsory:** Review of five research paper on the same research topic must be performed by an individual students and report must be submitted to the mentor. This is evaluative part of internal assessment. **All the papers must be selected from UGC care list for which mentor should help to the students.**

Refences

1. Vogels Textbook of Inorganic Quantitative Analysis, A. I. Vogel, 3rd Ed.
2. Standard methods of chemical analysis, Welcher
3. Lab Manual in biochemistry, immunology and biotechnology, Arti Nigam, Archana Ayyagari, Tat-McGraw-Hill Publication.
4. Indian Pharmacopeia
5. Green Chemistry Synthesis, Pawia
6. An introduction to Practical Bichemistry, David T. Plummer, Tata McGraw-Hill publishing Company Ltd.
7. Polymer Synthesis and Characterization, A Laboratory Manual, Stanely R Sandler, Wolf Karo, Jo-Anne Bonesteel, Eli M Pearce, Published by Academic press (Elsevier).
8. https://pubs.acs.org/doi/pdf/10.1021/ie50163a037, *Org. Synth.* **1922**, *2*, 47DOI: 10.15227/orgsyn.002.0047

CBOP-5, CHA-493: B) Project

a) At least 1/3 students of strength at M. Sc.-II must be allotted projects

b) Each student will perform project separately. Working hours are same as practical of CHA-493(A) project length should be sufficient and should be equivalent to 24 practical. Project report must be written systematically as: Front page, certificate, content, summary of project (2-3 page) followed by introduction (4 to 7 pages), literature survey (4-7) pages (recently published 20 to 30 papers must be included), experimental techniques, results, discussion and conclusions. If student is performing project in another institute, for such a student, internal mentor must be allotted and he will be responsible for internal assessment of a student. In this case student has to obtain certificate from both external and internal mentor. A project candidate must write note book date wise and systematically. **Note that** at the time external evaluation candidate should **submit his project note book** so that examiners can verify the results and systematic working of the students during project completion.

CCPP-4, CHA-494: Applied Analytical Chemistry

Sec-I: Analysis of Pharmaceuticals

Sr. No.	**Compulsory Practical**

1-4	Total analysis of aspirin as raw material as per Indian Pharmacopeia **except** limit test for arsenic (In assay part perform standardization of NaOH with potassium hydrogen phthalate). Express result as aspirin content ± Standard deviation. (Ref-1)
	Any 4 from 5-9
5	Tablet dissolution test on paracetamol Indian Pharmacopeia (Ref-1) or UV-absorbance based assay of plane paracetamol table using specific absorbance (British Pharmacopeia). (Ref-4)
6	Analysis of Ca- Gluconate 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-1)
7	Moisture content by i) Loss on drying of caffeine (oven drying method) and water content of dextrose (anhydrous or monohydrate) by Karl Fischer Method. (Ref-1 and 2)
8	Estimation of Benzocaine after extraction in chloroform by non-aqueous titration (Ref-3) or Estimation of Nicotinamide **or** caffeine by non-aqueous titration method according to IP (Ref-3) [standardize perchloric acid with potassium hydrogen phthalate]
9	Limit Tests for Fe, Ba and nitrate on dibasic calcium phosphate.
	Table Work (student self-activity): Analysis of IR spectra **for identification** of at least four pharmaceutical compounds from Indian Pharmacopeia or British Pharmacopeia (Spectrum from IP or BP can be used or you can record the IR spectra and analyse. (Ref-1, Ref-4)
	Any 4 from 9-14
9	Determination of NaCl (Cl by potentiometric titration or Na by flame photometry) and Dextrose (by polarimetry) in dextrose – sodium chloride type of saline solution. (Ref-1, 3).
10	a) Determination of refractive index of four liquids as per IP. b) Viscosity of ethyl cellulose by Oswald viscometer using viscometer which comply specification of IP.
11	The Determination of Aspirin and caffeine in a Proprietary Analgesic or given mixture by Ultraviolet (UV) Spectrometry. (Ref. – 8)
12	Analysis of Caffeine and benzoic acid from cold drink by HPLC (Ref-6, 9) **Or** HPLC Analysis of an Asthma Medication (Ref-7) **Or** Assay of Omeprazole in Gastro-Resistant Omeprazole Tablets (Solid Preparation) by LC (Ref.-6) **Or** Quantitative Determination of Methyl Paraben in a Prepared Sample by HPLC (Ref-9)
13	Kit method (any two): a) Analysis of glucose from blood or hydrolysed food sample and b) urea from urine, c) Cholesterol from blood or fatty material. d) Creatinine (Ref: Perform experiment as per the instructions of manufacturer of kit).
14	Visit to waste water treatment plant (industry or municipal corporation) and writing a detailed report on methods and parameters used for treatment process. Or

	Visit to Pharmaceutical Industry and report on function of QC department in pharmaceutical industry

Reference

1) Indian Pharmacopeia Volume I, 7th Ed
2) Indian Pharmacopeia Volume II, 7th Ed
3) Indian Pharmacopeia Vol-III, 7th Ed.
4) Introduction to Pharmaceutical Analytical Chemistry, Stig Pedersen-Bjergaard, Bente Gammelgaard, Trine Grønhaug Halvorsen, Second Edition, Wiley (2012).
5. Vogel's Textbook of Quantitative Chemical Analysis, 6th Ed.
6. Analysis of Soft Drinks: UV Spectrophotometry, Liquid Chromatography, and Capillary Electrophoresis, Journal of Chemical Education, Vol. 75 No. 5 May 1998
7. HPLC Analysis of an Asthma Medication, Journal of Chemical Education, Volume 85 Number 10 October 1988.
8. Experiments in modern analytical chemistry, D. Kealey, Springer Science Business media, 1986.
9. Analytical Chemistry for Technicians, John Kenkel, Third Edition, CRC Press LLC, 2003.

	Section-II
	Any four from 1-6
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)
	Any two from 7-10
7	Qualitative test for phosphate in hard water / soil sample /food / detergent and its estimation by colorimetry. (Ref-2, 3, 10-detergent)
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 of Cr(VI) by diphenyl carbazide method.
10	Demonstrating the Presence of Cyanide in Bitter Seeds while Helping **s**tudents Visualize Metal–Cyanide Reduction and Formation in a Copper Complex Reaction. (Ref.: 12,13)
11	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)
	Any two from 12-14
12	Electrochemical treatment to liquid waste (water soluble organics) (Ref-1, 4)
13	Photochemical remediation of pollutants (Ref-1)
14	Chemical mineralization of pollutants by Fenton's Process (Ref-1)

	Any two from 15-18
15	Vit-C in food / Lemon juice / or related juice by titration with 2-6 dichlorophenol indophenol (Ref-6, 7) **or** Estimation of Vitamin-C by reaction with Fe(III) and estimation of Fe(II) colorimetrically. (Ref-5)
16	Determination of total casein and lactose in milk [FSSAI Manual] (Ref.-8)
17	Saponification and iodine value of edible oil (Ref-6)
18	Adulteration Test for Milk and Milk product (Ref-8, 9)
	Any two from 19-24
19	Determination of molecular wt. of anionic detergent (Ref-10: 107-108, 120-121)
20	Determination Critical Micelle Concentration of detergent powder or pure detergent by conductometry / viscometry (Ref.-14)
21	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)
22	a) Molecular weight of polystyrene by viscometer b) Determination of water absorption by polymer (Ref-11)
23	Determination of chlorine content in PVC (Ref-12)
24	a) Determination of carbon black content in polymer b) Determination of swelling network in polymers (Ref-11)
	Students activity
12	Estimation of Glucose – Glucose in different samples can be analysed by i) titration with Fehling solution b) Titration with Iodine c) Colorimetry Folin-Wu method or DNSA method d) Colorimetry-Glucose by oxidase peroxidase method. Samples are – a) glucose in saline (DNS), b) glucose in urine / blood sample c) glucose in glucose supplement d) glucose in food. Give your choice of method for sample assigned to you by your mentor and analyse the sample. **Note:** Such many experiments can be designed by a mentor for internal evaluation of a student.

References

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.
4. Laboratory Experiments on Electrochemical Remediation of the Environment. Part 4: Color Removal of Simulated Wastewater by Electrocoagulation–Electroflotation, Journal of Chemical Education, Vol. 75 No. 8 August 1998
5. Vitamin C as a Model for a Novel and Approachable Experimental Framework for Investigating Spectrophotometry, Journal of Chemical Education, DOI:10.1021/acs.jchemed.9b00197

6. Biochemical Methods, Third Edition, By S Sadashivan, A. Manickam; New Age International publishers.
7. Lab. Manual: Manual of Methods of Analysis of Foods, Vegatables: Fruit and vegetable products: https://old.fssai.gov.in/Portals/0/Pdf/Draft_Manuals /FRUITS_AND_VEGETABLE.pdf
8. 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 Milk And Milk Products: https://old.fssai.gov.in/Portals/0/Pdf/Draft_Manuals/MILK_AND_MILK_ PRODUCTS.pdf
9. Common milk adulteration and their detection techniques, Azad and Ahmed International Journal of Food Contamination (2016) 3:22 DOI 10.1186/s40550-016-0045-3
10. Introduction to Surfactant Analysis, Edited by D. C. Cullum, Springer-Science + Business Media, B.V, 1994.
11. Experiments in polymer science, D. G. Hundawale, V. D. Athawale, V.R. Kapadi, V.V. Gite, New Age International Publishers.
12. Improved ninhydrin-based reagent for spectrophotometric determination of ppb levels of cyanide, Environmental Forensics, Volume 17, 2016 - **Issue 1**, **https://doi.org/10.1080/15275922.2015.1091404.**
13) Demonstrating the Presence of Cyanide in Bitter Seeds while Helping students Visualize Metal–Cyanide Reduction and Formation in a Copper Complex Reaction, *J. Chem. Educ.* 2016, 93, 5, 891-897.
14. Practical Physical Chemistry, Viswanathan B., Raghawan, Viva Books

Important Notes

1. For all three practical papers - ***Journal should be completed by the candidate on the same day before leaving of the lab***. *This is to avid manipulation of data by a student and to make habit of writing the experimental data and procedure systematically.* Chance should not be given to manipulate original data to the candidate. In fact, many students adjust or manipulate data from their lab work. If journal is completed before leaving the lab it will not encourage students to "adjust" the facts from their lab work to conform more closely to expectations from theory. (**Ref-Journal of Chemical Education, Min J. Yang and George F. Atkinson, Designing New Undergraduate Experiments, Vol. 75, No. 7, July 1998**). Higher weightage should be given in internal evaluation for the accuracy of the results.
2. ***Printed journal*** *consisting of name of experiment, special instructions regarding the safety precautions and special care to be taken (during handling of hazardous chemicals, electrodes, special glass apparatus, etc.), chemicals, apparatus, brief procedure and blank tables is allowed. It should* ***not contain*** *any details of calculations, dilutions factors, calculated amounts, reactions, and structures. Student has to write theoretical background, diagrams, structure, etc. at the end of experiment for which mentor should give sufficient time to the candidate.*
3. Wherever possible use / prepare minimum amounts / required amounts of solutions. Use micro burette for titrations involving instrumental methods. Micropipettes shall be used for measuring small volumes accurately which helpful to prepare small volumes of

solutions for instrumental analysis. For flame photometry / AAS typically 10 ml solution is sufficient, HPLC – 1-5 ml, colorimetry / spectrophotometry 5 ml, etc.

4. In colorimetric estimation do ***not prepare more than 5 ml solution*** for measurement of absorbance. Add all the reagent with micro-burette or 1 / 2 ml graduated pipette so that student will not require volumetric flask. If possible, use **1 ml cuvette** with spectrophotometers (It is available in market). Solvent extraction procedure can be typically performed within 2-ml to decrease toxic waste.
5. Develop micro or semi-micro methods for known /recommended procedures.
6. Avoid use of toxic chemicals and reagents and if possible, replace toxic reagent by non-toxic or less toxic reagent. Example: in volumetric estimation of Fe(III) $SnCl_2$ and then $HgCl_2$ is used to convert Fe(III) to Fe(II). Sn(IV) and Hg(I) produced in reaction are toxic. This can be done by using Zn metal powder. Avoid use of $K_2Cr_2O_7$, and no alternative prepare minimum /required amount of it.
7. **Important Note:** Wherever required, s***tandardize $Na_2S_2O_3$ with oven dried KIO_3 in place of $K_2Cr_2O_7$ as Cr(VI) is carcinogenic and mutagenic.***
8. By trial replace $CHCl_3$ by other extracting solvents as chlorinated solvents are highly toxic.
9. Metal like Ag can be recovered after experiment. Device suitable method.
10. Wherever possible replace calomel electrode by Ag/AgCl reference electrode as Calomel consists of toxic element Hg and Hg(I). (**Ref-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).**
11. College / Chemistry Department of the respective college must follow all the rules of EPA / WHO regarding the toxic waste management of the chemistry laboratory produced during practical.
12. In each practical course a mentor can introduce one or two **Novel** experiment of analytical chemistry. Experiment should be equivalent to one practical session (4 h duration) or two practical sessions (long experiment). For such replacement students can be exempted one or two regular experiment respectively. A teacher can promote to a student for such Novel analytical chemistry experiments provided that department is ready to support such experiment. The newly introduced experiment will the inherent part of external evaluation. Example-identification and estimation of melamine from milk powder, pesticide residue from vegetables, estimation of As(III) from bore well water, synthesis / extraction of novel organic compound and its total spectral characterization, etc.