VIVEKANAND COLLEGE, KOLHAPUR
(EMPOWERED AUTONOMOUS)

DEPARTMENT OF CHEMISTRY
Two Years PG Programme

Curriculum, Teaching and Evaluation Structure

for

M. Sc. – II

Analytical Chemistry

Semester – III & IV

(Implemented from academic year 2023 - 24 onwards)

Vivekanand College, Kolhapur (Autonomous)

Department of Chemistry

M. Sc. Part-II, (Sem III and IV)

Analytical Chemistry Syllabus (CBCS) 2023-24

Total No. of Semester – 02

Total No. of Papers – 08

No. of papers (theory) per semester – 04

No. of practical course per semester – 02

Maximum marks per paper (practical) -100

Distribution of Marks – Internal evaluation - 20

External evaluation - 80

(Semester exam.)

Total Marks for M. Sc. Degree

Theory Paper: 1600

Practical course: 800

Total: 2400

M. Sc. Part - II, (Sem - III & IV) Analytical Chemistry

SEMESTER- III

Paper No. – IX	: Advanced Analytical Techniques
Paper No. – X	: Organo Analytical Chemistry
Paper No. – XI	: Electroanalytical Techniques in Chemical Analysis

ELECTIVE PAPERS

Paper No. – XII (A)	: Environmental Chemical Analysis and Control
Paper No. - XII (B)	: Recent Advances in Analytical Chemistry
Practical Course	**: Practical-V and Practical-VI**

SEMESTER- IV

Paper No. – XIII	: Modern Separation Method in Analysis

Paper No. – XIV	: Organic Industrial Analysis
Paper No. – XV	: Advanced Methods in Chemical Analysis

ELECTIVE PAPERS

Paper No. – XVI (A)	: Industrial Analytical Chemistry
Paper No. – XVI (B)	: Quality Assurance and Accreditation
Practical Course	**: Practical – VII and Practical-VIII**

VIVEKANAND COLLEGE KOLHAPUR (Empowered Autonomous) ESTD JUNE 1964

Vivekanand College (Autonomous), Kolhapur
M. Sc. Part - II (Analytical Chemistry)
CBCS Syllabus with effect from June - 2023
Semester - III
Paper No. - IX: Advanced Analytical Techniques
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to:

CO1: Learn mass spectrometry by outlining its principles, historical context, compound-based classification, ionization techniques, and diverse mass analyser comparisons.

CO2: Understand the importance of nanomaterials and nanotechnology, distinguish between diverse nanomaterial dimensions, elucidate synthesis methods with examples, and evaluate application areas across fields.

CO3: Attain expertise in advanced microscopy techniques by comprehending working principles, recognizing practical applications, and distinguishing between methods based on strengths and limitations.

CO4: Gain comprehensive proficiency in a spectrum of spectroscopic techniques, encompassing principles, instrumentation, data analysis and practical applications.

UNIT-I: Advances in Mass Spectrometry **(15)**

Introduction to Mass spectrometry, diagram of a mass spectrometer and Instrumentation, principles, history, concept of ion free path, classification of mass spectrometry based on nature of compound to be analyzed and the ion sources viz. Electron impact (EI), chemical ionization (CI), Fast ion or atom bombardment ionization (FID/FAB), field desorption (FD), laser desorption ionization (LDI), plasma desorption ionization (PDI), thermospray ionization (TSI), electrospray (ESI), atmospheric pressure ionization, Inductively couple plasma (ICP) etc. Mass Analyzers, Quadrupolar Analyzers, Quadrupole ion trap or Quistor, Ion trap detector, development of high –Mass, High-resolution ion trap, tandem mass spectrometry in the ion trap, time of flight analyzer, magnetic and electromagnetic analyzer, ion cyclotron resonance and FT-MS, and detectors.

UNIT-II: Introduction to Nanotechnology and Nano Chemistry **(15)**

Definition of nanomaterials and nanotechnology, significance of nanotechnology, size and properties, types of nanomaterials like 0D (quantum dots), 1D, 2D and 3D, introduction to physical, chemical and biological synthesis of nanomaterials with suitable examples, top down and bottom-up approach, chemical synthesis of nanomaterials - Different types and processes for

synthesis of nanomaterials using wet chemical approaches. Fabricating nanomaterials with different morphology intended for specific applications, Applications of Nanotechnology.

UNIT-III: Advanced Instrumentation Techniques-A **(15)**

Scanning Electron Microscope (SEM) - Introduction, principle, instrumentation, applications

Transmission Electron Microscope (TEM) - Introduction, principle, instrumentation, applications

Electron Dispersion Spectroscopy (EDS) - Introduction, principle, instrumentation, applications

Energy Dispersive X-ray Analysis (EDAX) - Introduction, principle, instrumentation, applications

Scanning Tunneling Microscopy (STM) - Introduction, principle, instrumentation, applications

Atomic Force Microscopy (AFM) - Introduction, principle, instrumentation, applications

Practical applications and examples in analytical chemistry and research.

UNIT-IV: Advanced Instrumentation Techniques-B **(15)**

Raman Spectroscopy- Introduction, principle, instrumentation, applications

X-Ray Fluorescence Spectroscopy (XFS) - Introduction, principle, instrumentation, applications

Electron Spin Resonance Spectroscopy (ESR)- Introduction, principle, instrumentation, applications

X-Ray Photoelectron Spectroscopy (XPS)- Introduction, principle, instrumentation, applications

Auger Electron Spectroscopy - Introduction, principle, instrumentation, applications

Secondary Ion Mass Spectrometry (SIMS)- Introduction, principle, instrumentation, applications

Practical applications and examples in analytical chemistry and research.

Recommended Books:

1) E. De. Hoffmann, J. Charette, V. Stroobant, Mass Spectroscopy: Principles and Applications, John Wiley & Sons, Masson, Paris 1996.
2) J. H. Gross, Mass Spectroscopy: A Text book, Springer-Verlag Berlin 2004.
3) C. G. Herbert, R. A. W. Johnstone, Mass Spectrometry Basics, CRC Press, Boca Raton, Florida, 2002.
4) K. Benjamin : Mass Spectrometry
5) A. I. Vogel: A text book of Quantitative inorganic Analysis, Lonqmans.
6) G. H. Morrison and H, Freiser : Solvent Extraction in Analytical Chemistry (John Wiley New York, 1958)
7) Willard, Merrit and Settle: Instrumental Methods of analysis.

8) Principles of instrumental analysis- Holler, Skoog and Crouch
9) Instrumental methods of Chemical analysis - H. Kaur
10) Bhushan, Bharat 2004. Handbook of Nanotechnology. Springer.
11) Niemeyer, C.M. & Mirkin, C.A. 2004. Nanobiotechnology- Concepts, Applications and Prespectives. Wiley-VCH Verlag.
12) Zander, C., Enderlein, J. & Keller, R.A. 2002 Single Molecule Detection in Solution. Wiley-VCH Verlag.
13) Avouris, P, Klitzing, K. Von, Sakaki, H. & Wiesendanger, R.2003 NanoScience and Technology.
14) Series. Scanning Probe Microscopy- Analytical Methods (R. Wiesendanger eds), Springer.
15) Instrumental Analysis by Skoog
16) Nanochemistry, a chemical approach to nanomaterials, G. A. Ozin, and A. C. Arsenault, RSC Publishing, Cambridge, 2005. ISBN 0-85404-664-X.

Paper No. - X: Organo Analytical Chemistry
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to:

CO1: Develop a profound grasp of UV-Visible, IR, 1H-NMR, 13C NMR, and Mass Spectrometry, encompassing fundamental principles, advanced organic analysis, and the ability to solve intricate structural determination challenges.

CO2: Proficiently classify drugs, pinpoint potential impurity sources in pharmaceutical raw materials, execute limit tests for Pb, As, Fe, and utilize a range of analytical techniques, including UV, colorimetric, and biological assays, for the comprehensive analysis of drugs and vitamins.

CO3: Gain a comprehensive knowledge of clinical analysis and enzyme assay techniques, showcase adeptness in collecting, preserving, and analysing physiological fluids, conduct estimations of key constituents in blood and urine samples, and effectively employ analytical methods for diagnosing diseases.

CO4: Engage in a comprehensive exploration of pesticide introduction, classification, and analysis, utilizing colorimetric and chromatographic techniques like GC-MS, HPLC-MS to examine pesticide residues, and the estimation of toxic substances such as lead, mercury, arsenic in biological samples for forensic applications.

UNIT–I: Hyphenated Techniques **(15)**

Advanced techniques of analysis: UV-Visible, IR, 1H-NMR (Recapitulation), 13CNMR, Mass spectrometry (Basic fundamentals of mass spectrometry, ionization, advanced organic analysis examples); Problems related to structure determination and applications of spectroscopic techniques as analytical tools.

UNIT-II: A) Drug Analysis **(10)**

Introduction to drugs, their classification, sources of impurities in pharmaceutical raw materials such as chemical, atmospheric and microbial contaminants etc. Limit tests: Limit test for impurities for Pb, As, Fe, Se, etc. Estimation of moisture (K-F method), halide (Schnoiger's oxygen flask method), sulfate, boron, etc. Analysis of commonly used drugs such as antihistamines, sulfa drugs, barbiturates, etc. using non-aqueous titrations, sodium nitrite titrations, differential UV methods, colorimetric and fluorimetric methods of analysis.

B) Analysis of vitamins **(05)**

Analysis of vitamins (thiamine, ascorbic acid, Vit. A, Vit. B6, Vit. K) and hormones

(progesterone, oxytocin, insulin) chemical, instrumental and biological assay, wherever applicable.

UNIT–III: A) Clinical Analysis **(08)**

Biological significance, analysis of assay of enzymes (pepsin, monoamine, oxidase, tyrosinase), Composition and detection of abnormal level of certain constituents leading to diagnosis of diseases. Sample collection and preservation of physiological fluids, analytical methods to the constituents of physiological fluids (blood, urine and serum). Blood- Estimation of glucose, chlolesterol, urea, hemoglobin and bilirubin, Urine- urea, uric acid, creatinine, calcium, phosphate, sodium, potassium and chloride.

B) Body fluid analysis **(07)**

Composition and detection of abnormal level of certain constituents leading to diagnosis of diseases. Sample collection and preservation of physiological fluids, analytical methods to the constituents of physiological fluids (blood, urine and serum) Blood-Estimation of glucose, chlolesterol, urea, hemoglobin and bilirubin Urine- urea, uric acid, creatinine, calcium, phosphate, sodium, potassium and chloride.

UNIT–IV: A) Pesticides Analysis **(07)**

Introduction, classification of pesticides, sampling, sample pretreatment and processin g, analysis of DDT, gammexane, endosulphan, zinab, ziram, malathion, thiram, thiometon, simazine and chloridane. Applications of colorimetric and chromatographic techniques (GC-MS, HPLC-MS) in analysis of pesticide residue. Introduction to EPA regulatory body. Practical applications and examples in analytical chemistry and research.

B) Forensic Analysis **(08)**

Special features of forensic analysis, sampling, sample storage, sample dissolution, classification of poisons, lethal dose, significance of LD-50 and LC-50. General discussion of poisons with special reference to mode of action of cyanide, organophosphate and snake venom. Estimation of poisonous materials such as lead, mercury and arsenic in biological samples.

Practical applications and examples in analytical chemistry and research.

Reference Books:

1) F. J. Welcher: Standard methods of Chemical analysis, 6th Ed. Vol. I and II (D. Van Nostard Comp.)
2) I. M. Kolthoff: Treatise on Analytical Chemistry Vol. I & II

3) F. D. Snell: Encyclopedia of industrial Chemical Analysis Vol. 1 to 20 (John Wiley)
4) Riech: Outline of Indutrial Chemistry.
5) K. H. Buchel: Chemistry of Pesticides (John Wiley)
6) Indian, Pharmacopoeia, British Pharmacopoeia and U. S. Pharmacopoeia.
7) V. M. Parikh: Absorption spectroscopy of organic molecules (Addision Wesley)
8) Willard, Merrite, Dean and Settle: Instrumental methods of analysis (CBS)
9) D. H. Williams and J. Fleming: Specroscopic methods in organic chemistry (Mc Graw Hill)
10) Silverstein: Spectroscopic Identification of organic compounds (John Wiley)
11) Jackmann and Sternhill: Applications of NMR spectroscopy of organic Chemistry (Pergamon Press)
12) J. D. Roberts : Nuclear Magnetic Resonance (Mc Graw Hill)
13) K. Benjamin : Mass Spectrometry
14) Nichollas: Aids to the Analysis of foods and Drugs.
15) A. H. Beckett and J. B. Stanlake; Practical Pharmaceutical Chemistry Vol. I & II (CBS publishers)
16) S. Ranganna: Handbook of analysis and quality control for fruits and vegetable products (McGraw Hill)
17) Ramalu: Analysis of pesticides

Paper No. - XI: Electroanalytical Techniques in Chemical Analysis
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to:

CO1: Develop a comprehensive comprehension of voltammetry principles, encompassing cyclic voltammetry, pulse voltammetry, and stripping voltammetry, for analyte determination and showcasing their real-world applications in analytical chemistry and research.

CO2: Understand the classification, properties, theories of charge origin, stability, factors influencing coagulation and spontaneous ageing of colloids, as well as differentiate types of emulsions, elucidating their preparation, properties, and demulsification methods.

CO3: Attain a thorough understanding of particle size analysis methodologies such as LASER light scattering, dynamic light scattering, and photosedimentation, including theoretical models like Mie theory and Fraunhofer diffraction theory and to apply diverse techniques like XRD, SEM, and TEM for real-world particle size measurements.

CO4: Explain different ion-selective electrode types, encompassing glass, solid-state, liquid-liquid membrane, enzyme, and gas electrodes, detailing their construction and applications.

UNIT-I: Voltammetry Techniques **(15)**

Introduction, Principle, excitation signals in voltammetry, basic instrumentation based on operational amplifiers, voltammetric electrodes

Cyclic Voltammetry: Instrumentation, Determination of analytes using cyclicvoltammetry, Applications.

Pulse voltammetry: Introduction, Normal Pulse Voltammetry, Reverse pulse voltammetry, Differential pulse voltammetry, Square wave voltammetry.

Stripping voltammetry: Cathodic and Anodic stripping voltammetry, Electrodeposition step, Voltammetric completion of the analysis, adsorptive striping methods, voltammetry with microelectrodes. Practical applications in analytical chemistry and research.

UNIT-II: **(15)**

a) Ion selective electrodes & Electrochemical sensors:

Introduction, types and construction of electrodes, glass electrode, solid state and precipitate electrodes, liquid – liquid membrane electrodes, enzyme and gas electrodes, Chemically modified electrode, Enzyme based electrode, catalytic electrodes, ultramicroelectrodes and applications.

b) Electrogravimetry

Introduction, Types of electrogravimetric techniques, Diffusion Migration, Convection, instrumentations, applications.

UNIT–III: Particle Size Analysis **(15)**

Introduction, Low angle LASER light scattering: Instrumentation, theoretical models, Mie theory, Fraunhofer diffraction theory, particle size distribution analysis, Applications. Dynamic Light Scattering: Introduction, Instrumentation, photodetector sample cell and sample handling, Applications, Photosedimentation: Setting velocity and particle size, Stokes equation, Instrumentation, sedimentation modes, Particle size distribution analysis, photometric measurements and applications. Comparison with particle size measurements using XRD, SEM and TEM. Practical applications in analytical chemistry and research.

UNIT–IV: Electrophoresis: **(15)**

Introduction, Paper electrophoresis Principle, Factors governing migration of ions, Supporting media (gel, paper, cellulose, acetate, starch, polyacrylamide, agarose, sephedax and thin layers) Techniques of electrophoresis: Low and high voltage, iso electric focusing, continuous electrophoresis, capillary electrophoresis, Zone, gel, isotaechophoresis and miceller electro kinetic capillary chromatography, instrumentation, detection and applications and Applications, Numericals.

Reference Books:

1) R.D. Braun, Introduction to Instrumental Analysis.
2) D.A. Skoog, F. J. Holler, Principles of Instrumental Analysis, 6th edition.
3) Willard, Deritt, Dean and Settle, Instrumental methods of Analysis.
4) F. J. Welcher, Standard Methods of chemical Analysis Vol.3, Part A & B.
5) G.W. Ewing, Instrumental Methods of Analysis 4th and 5th editions.
6) Chatawal and Anand, Instrumental Methods of Analysis.
7) Bassett, Denney-Jeffer and Mendham, Vogel's Textbook of Quantitative Inorganic Analysis, (5th edition).
8) Electro-analytical chemistry, edited by H.W. Nurnberg.
9) Stulic, Ion selective electrodes (John Wiley).
10) Introduction to instrumental analysis by R. D. Broun, Mc Graw Hill (1987)

11) Instrumental methods of chemical analysis by H. Willard, D. Merrit, J.A. Dean and F.A. Settle. Sixth edition CBS (1986)

12) Fundamentals of Analytical Chemistry by D. A. Skoog, D. M. West and H. J. Holler sixth edition (1992) and Principles of Instrumental Analysis Skoog, West, Niemann

13) Vogel Text Book of quantitative analysis 6th Ed.

Elective Papers
Paper No. – XII (A): Environmental Chemical Analysis and Control
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to

CO1: Develop a comprehensive grasp of sampling theory, techniques, and criteria for gases, liquids, and solids; implement strategies to minimize variables during sampling and ensure efficient transmission and storage of collected samples.

CO2: Acquire expertise in performing comprehensive environmental analyses through a range of electrochemical techniques, including conductometry, potentiometry, cyclic voltammetry, amperometry, and coulometry, fluorescence spectrometry, inductively coupled plasma spectrometry, turbidimetry, and non-dispersive infrared analysis (NDIR) for diverse environmental applications.

CO3: Develop a comprehensive understanding of the chemistry, sources, characterization, and analysis methods for air pollutants, major minor components in potable and industrial water, conducting measurements for parameters including DO, COD, and BOD; and perform in-depth analyses of pollutants such as Pd, Cd, Hg, Cr, As in water, including quality assessments of organic and inorganic constituents in industrial wastewater.

CO4: Grasp the lifecycle of phenolic residues, including their sources, disposal, treatment, and analysis, with a focus on recovery methods from liquid effluents; adeptly conduct analysis of organomercurials, organochlorine pesticides, and volatile organic pollutants, employing suitable analytical methods.

UNIT-I: Sampling in analysis **(15)**

Definition, theory and techniques of sampling, sampling of gas, liquids and solids, Criteria of Good sampling, Minimization of Variables, transmission and storage of samples, high pressure ashing techniques (HPAT), particulate matter, its separation in gas stream, Filtering and gravity separation. Analysis of particulate matter like asbestos, mica, dust and aerosols etc

UNIT-II: Electrochemical and spectral methods Environmental analysis **(15)**

Introduction to instrumental techniques, principle instrumentation and applications with respect to environmental analysis of Conductometry, Potentiometry, Ion selective electrodes, Amperometry, Coulometry, Atomic absorption spectrometry, Atomic fluorescence spectrometry, Inductively coupled plasma spectrometry, Turbidimetry, Non Dispersive Infrared Analysis (NDIR).

UNIT-III: Air and Water Pollutant Analysis **(15)**

Chemistry of Air pollutants, characterization. source, methods of analysis of air pollutants; CO, CO_2, NO_X, NH_3, H_2S, SO_2 etc. Monitoring Instruments, Potable and Industrial water, major and minor components, dissolved oxygen (DO) Chemical oxygen demand(COD) Biochemical oxygen demand (BOD) and their measurements. Analysis of Pd, Cd, Hg, Cr, As and their physiological manifestations. Quality of industrial waste water analysis for organic and inorganic constituents. Chemistry of odour and its measurements.

UNIT-IV: Organic Pollutants and Their Analysis **(15)**

Sources, disposal, treatment and analysis of phenolic residues, methods of recovery of phenols from liquid effluents, Organomercurials and its analysis, Analysis of organochlorine pesticides, volatile organic pollutants and their analysis

Recommended books:

1) A.K. De: Standard Methods of Waste and Waste water analysis.
2) P. M. S. Monk Fundamentals of Electroanalytical chemistry-John Wiley & Sons (2001) 3. Instrumental methods of chemical analysis H. Kaur
3) S.M. Khopkar, Environmental Chemistry ; Environmental pollution analysis
4) M.S. Creos and Morr, Environmental Chemical Analysis, American publication (1988)
5) A.K. De, Environmental Chemistry, New Age International publishers. Moghe and Ramteke, Water and waste water analysis: (NEERI)
6) A.C. Stern, Air pollution: Engineering control Vol. IV(AP)
7) P.N. Cheremisinoff and R.A. Young, Air Pollution control and Design. Hand Book Vol. I & II (Dekker)
8) R.B. Pohasek, Toxic and Hazardous waste disposal, Vol.I& II (AAS) 9)M.Sitting, Resources Recovery and Recycling, Handbook of industrial Waste. 10)B.K.Sharma, Industrial Chemistry.
11) S.P. Mahajan, Pollution Control in Process Industries.
12) R.A. Horne, Chemistry of our Environment.

Paper No. – XII (B): Recent Advances in Analytical Chemistry
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able

UNIT-I: Ultra Purity and Ultra trace Analysis **(15)**

Ultra purity and ultra trace analysis, laboratory dosing, purification of reagents, Preconcentration Techniques, Methods of trace analysis such as NAA, XRF, AAS and ICP, High purity materials for electronic industry, contamination control during analytical operations.

UNIT-II: Radio-analytical Chemistry **(15)**

Separation methods, Precipitation, solvent extraction and chromatographic methods. Activation analysis, basic principles, fast neutron activation analysis, radiochemical methods in activation analysis, Applications if Geo-chemistry, oxygen in metals. Isotope dilution analysis: Principles and applications. Sub-stoichiometric determination of traces of metals: Principles, techniques and experimental methods in the determination of As, Pb and Hg.

UNITS-III: Advanced Techniques in Analysis **(15)**

Nuclear Magnetic Resonance Spectroscopy (^{1}H NMR):

Elementary ideas (Recapitulation); Different types of couplings, factors affecting on coupling constants, Karplus equation, Spin systems (AB, AX, ABX, AMX), Rate processes, spin decoupling, shift reagents, Nuclear Overhauser effect (NOE), INEPT and INADEQUATE.

^{13}C Nuclear Magnetic Resonance Spectroscopy

Elementary ideas, instrumental problems, chemical shifts (aliphatic, olefinic, alkyne, aromatic, heteroaromatic and carbonyl carbons); Effect of substituents on chemical shifts.

UNIT-IV: Electron Spin Resonance Spectroscopy **(15)**

Electron behavior, ESR spectrometer, Spectra, Hyperfine interaction, free radical and interpretation of the spectra, Applications in quantitative analysis. Numerical problems.

Recommended books:

1) Garen W. Ewing, Analytical Instrumentation, Handbook, Marcel Dekker Inc. (1997).
2) Mereitt, Dean, Settel, Instrumental methods of Chemical Analysis.
3) M. Zeif and J.W.Mitchell, Contamination Control in trace elemental analysis.
4) Ajuja, Ultrapurity.

5) Minczewski, Chwastowska and Dycozynski, Separation and pre-concentration methods in Inorganic trace analysis. Ellis Haward.
6) Cali, trace Analysis of semiconductor Materials Pergamon.
7) Overman and Cleark, Radioisotopes techniques MGH.
8) Tolgyessy, Brown and Kyrs, Isotope dilution analysis.
9) Leniham and Thomson, Activation Analysis(AP)
10) Ruzica and Stary, Substopchiometry in Radiochemical Analysis. Pergamon.
11) Ladd and Lee, Radiochemistry.
12) Clerk, Handbook of Radiochemical methods
13) Price, Nuclear radiation detections.

M. Sc. Part - II (Semester - III) Analytical Chemistry
Practical Course -V and VI

List of Experiments:

Major:

1. Estimation of Sn, Zn, Cu and Pb from Bronze alloy (volumetric, gravimetric or colorimetric techniques can be used)
2. Estimation of Ca and Fe from milk powder
3. Analysis of Galena ore
4. Analysis of Benzoic acid and salicylic acid from medicated powder
5. Analysis of vitamin A in food products
6. Estimation of Aspirin
7. Kjeldahl's method of protein estimation in foods and feeds
8. Analysis of Lindane in BHC powder.
9. Determination of pK value of an indicator.
10. Polarographic estimation of traces of Cu, Cd, Ni, Zn and Fe in sample solution.
11. To study the complex formation between Fe(III) and salicylic acid and determine the stability constants of the complex by Job's variation method.
12. To determine the equivalence conductance and dissociation constant using Kolharach Law at infinite dilution independent of ionic mobility of weak electrolyte.
13. Any other suitable experiment may be added when required.

Minor:

1. Analysis of plaster of Paris for calcium content
2. Fertilizer analysis for P (colorimetrically), K (Flame photometrically).
3. Determination of Barium ions by Turbidimetry.
4. Analysis of iodized table salt.
5. Analysis of soda ash.
6. Estimation of copper fungicide
7. Analysis of sulpha drug
8. Analysis of vitamin-C in juices and squashes.
9. Analysis of ethambutol

10. Identification of organic compounds by their IR spectra
11. Determination of strength of acetic acid in commercial vinegar by conductometric method.
12. Determination of chloride content from saline water by potentiometry.
13. Estimation of bicarbonate and carbonate by potentiometric method.
14. Estimation of Fe by ceric sulphate and potassium dichromate titration potentiometrically.
15. XRD and Thermal analysis Kaolinite, cobalt oxalate and zinc oxalate.
16. Estimation of vitamin B2 in the medicinal tablets fluorimetrically.
17. Kinetic study of hydrolysis of ethyl acetate in presence of OH – ions conductometrically.
18. Determination of pK of given dibasic acid pH-metrically.
19. Determination of relative strength of acetic acid, chloroacetic acid and trichloro acetic acid by conductometrically.

(At least 10 major and 10 minor experiments should be carried out)

Vivekanand College (Autonomous), Kolhapur
M. Sc. Part - II (Analytical Chemistry)
CBCS Syllabus with effect from June - 2023
Semester - IV
Paper No. - XIII: Modern Separation Methods in Analysis
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to:

CO1: Understand the advanced gas and liquid chromatographic techniques. They will be well-versed in the principles, instrumentation, working mechanisms.

CO2: Utilize hyphenated techniques, such as gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS), to enhance compound identification and quantification.

CO3: Apply their knowledge to practical scenarios and research projects. They will possess the skills to select appropriate chromatographic techniques based on the characteristics of analytes, separation requirements, and analytical objectives.

CO4: Grasp the modern extraction and separation techniques, including solid-phase extraction, solid-phase microextraction, sonic extraction, and accelerated solvent extraction.

UNIT-I: Advanced Gas Chromatographic Techniques **(15)**

Principles, Plate theory, Instrumentation and working of a Gas Chromatograph, sampling, sample pretreatment, sample injection types, columns, Detectors, programmed temperature G.C., Applications. Pyrolysis gas and vapour phase chromatography-instrumentation and techniques, advantages and applications. Gas chromatography-Mass Spectrometry, interface, instrumentation and applications. Introduction to TGA-MS/TGA-GC-MS and significance. Practical applications and examples in analytical chemistry and research.

UNIT-II: Advanced Liquid Chromatographic Techniques **(15)**

High Performance Liquid Chromatography (HPLC) and Ultra Performance Liquid Chromatography (UPLC)-Principle, instrumentation, mobile phase, Stationary support in HPLC, detectors and applications. Super critical fluid chromatography (SCFC), characteristics, instrumentation and applications. Comparison of HPLC and GLC with SCFC. Liquid

Paper No. - XIV: Organic Industrial Analysis
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to:

CO1: Understand the various chemical analysis techniques used in the industrial context.

CO2: Perform quality control analyses and ensure compliance with regulations in various industries. They will be capable of analyzing raw materials, additives, and finished products to assess their composition, purity, and adherence to industry standards.

CO3: Understand the principles of various techniques such as chromatography, spectrophotometry, titration, and bomb calorimetry.

CO4: Develop problem-solving skills to address issues related to contamination, adulteration, impurity identification, and quality assurance.

UNIT–I: Industrial Analysis

A) Analysis of oils, fats and Soaps **(08)**

Introduction to natural fats and oils; isolation of oils from natural resources and their purification. Analysis of oils and fats: Softening point, Congeal point, Titre point, Cloud point, Iodine, saponification, acid, hyroxyl, R-M and Polenske value, Elaiden test, etc.

Introduction to soaps, manufacture of soaps (in brief), analysis of soaps: total anhydrous soap and combined alkali, potassium, water, free fatty acids, saponifiable and non-saponifiable matter in soaps, estimation of phenol, copper and germicidal agents in soaps, determination of inorganic fillers and soap builders, and other additives, estimation of soap in detergents (THAM method)

B) Analysis of Detergents **(07)**

Classification of detergents, analysis of raw materials, separation as alcohol soluble and alcohol insoluble matter, additives in detergent formulation (chlorides, sulfates, phosphates, silicates, borates, oxygen releasing substances, CMC, EDTA, etc.), their role and analysis; analysis of active ingredients in detergents (methylene blue and Hyamine-1622 method).

UNIT – II: Food and Food Additive Analysis

A) Food Analysis **(08)**

Food flavors, food colors, food preservatives, analysis of milk and milk products, adulterants in

Chromatography-Mass Spectrometry interface, instrumentation, advantages and applications. Practical applications and examples in analytical chemistry and research.

UNIT-III: Ion Chromatography **(15)**

Principles, structure and characteristics of resins, eluent, supressor columns and detectors used in Ion Chromatography, commercial scope, analytical applications, environmental speciation by Ion Chromatography. Practical applications and examples in analytical chemistry and research.

UNIT–IV: A) Modern extraction and separation techniques **(08)**

Basic principles, classification of solvents extraction systems, extraction equilibria, factors affecting extraction process , application of β- diketones , δHydroxyquinoline , dithicarbamaes , xanthenes , Thio, separation of non metals and metals. Separation of transition metal ions using ion exchangers. Solid phase extraction, solid phase microextraction, sonic extraction, accelerated solvent extraction, soxhlet extraction.

B) Extractive Chromatographic Separations **(07)**

Introduction, Theoretical aspects of extraction chromatography, solvent extract extraction chromatography with chelating ligands, extracion chromatography by ion pair formation, extraction chromatography by solvation, extraction equilibria, nature of stationary phase in extraction chromatography, inert support, techniques in extraction chromatography, extraction chromatography with tributyl phosphate and other applications. Practical applications and examples in analytical chemistry and research.

Recommended Books:

1) A.I. Vogel, a text Book of Quantitative Inorganic Analysis.
2) W H Willard, L L Merritt and J A Dean, Instrumental Methods of Analysis.
3) S. M. Khopkar, Basic Concepts in Analytical Chemistry.
4) LR. Shyder and C.H. Harvath, An Introduction to separation Science. Wiley Interscience.
5) James S Fritz and George H. Schenk Jr. Quantitative Analytical Chemistry, 2nd editions Allyn and Bacon Inc. Bosten.
6) J.G. Dick, Analytical Chemistry.
7) R.L. Pescok and L.D. Shield, Modern Methods of Chemical Analysis.

8) O. Samuelson : Ion Exchange separation in analytical chemistry (Jhon wiley , 1963)

9) Y. Marcus and A. S. Kertes : Ion Exchange and solvent Extraction of metal complexes (Wiley – Interscience , 1969)

10) J. A. Marinsky and Y. Marcus : Ion exchange and solvent Extraction (Marcel Dekker, INC , New York, 1973)

11) G. H. Morrison and H, Freiser : Solvent Extraction in Analytical Chemistry (John Wiley, New York, 1958)

12) A. K. Da, S. M .Khopkar and R. A. chalmers :solvents Extraction of metals (Von Nostrant Ravinhold, 1970)

milk and their identification, analysis of honey, jam and their major component. Practical applications and examples in analytical chemistry and research.

B) Food Additive Analysis **(07)**

Additives in animal food stuff: Antibiotics: penicillin, chlorotetracyclin, oxytetracyclin in diet supplements; Identification and estimation of growth promoting drugs such as. sulfaquinoxaline, methyl benzoquate, sulfanitran, pyrimethamine, nitrovin, nitrofurazone, acinitrazole, etc

UNIT-III: Analysis of cosmetics products **(15)**

Introduction to cosmetics, definition, types of cosmetics, background, development in cosmetic industry, issues in cosmetic industries (contamination and adulteration), future scope and role of analytical chemistry.

A) Analysis of cream and lotions **(08)**

Composition of creams and lotions, determination of water, propylene glycol, non-volatile matter and ash content; estimation of borates, carbonates, sulphates, phosphates, chlorides, ammonia, nitromethane, oxalic acid, 4- hydroxy benzoic acid, sodium iodate, free formaldehyde, H_2O_2, mercatoacetic acid, titanium and zinc oxides. Practical applications and examples in analytical chemistry and research.

B) Analysis of face powder **(07)**

Composition of face powder, estimation of boric acid, Mg, Ca, Zn, Fe, Al and Ba. Analysis of deodorants and antiperspirants-composition, analysis of fats and fatty acids, boric acid, magnesium, calcium, zinc, iron, titanium, aluminium, phenol, methanamine, hexachlorophenone, sulphonates, urea, etc. Practical applications and examples in analytical chemistry and research.

UNIT-IV: Analysis of Paints, pigments and petroleum products

A) Analysis of Paints and pigments **(08)**

Composition of paint, preliminary inspection of sample, test on the total coating, separation and estimation of pigments, binder and thinner of latex paints; modification of binder, flash point of paints. Practical applications and examples in analytical chemistry and research.

(B) Analysis of petroleum products **(07)**

Introduction, constituents and petroleum fractionation, quality control; - specific gravity,

viscosity, Cloud point, pour point, flash point, vapor pressure, Doctor test, sulphuric acid absorption, aniline point, and colour détermination, cloud point, pour point. Determination of water, neutralization value (acid and base numbers),ash content, sulphur and mercaptan sulphur. Determination of lead in petroleum; Analysis of coal and coke: Types, composition, preparation of sample, proximate and ultimate analysis calorific value by Bomb Colorimetry.

Reference Books:

1) S. R. Junk and H. M. Pancoast: Hand book of sugars (AVI)
2) B. Bilot and B. V. Well: Perfumary technology (JW)
3) I. M. Kolthoff: Treatise on Analytical Chemistry Vol. I and II
4) D. Pearson: Laboratory techniques in food analysis.
5) S. Ranganna: Handbook of Analysis and Quality control for fruits and vegetable products, 2nd Ed. (Mc Graw Hill.)
6) Nicholls: Aids to the analysis of foods and drugs.
7) G. J. Mountrey: Poultry product technology (AVI)
8) Karamer Twig: Quality control for food industry (AVI)
9) G. F. Longonan: the analysis of detergents and detergent products (JW)
10) A. Davidsohn & B. M. Mlwidaky : Synthetic detergents (Book center, Mumbai)
11) M. Ash and L. Ash: A formulary of cosmetic preparations. (G. Goodwin)
12) Kurl Bauer, Dorothea Garhe, Horst Surburg: Common fragrance and flavour materials, (VCH publisher, New York)
13) F. J. Welcher: Standard Methods of Chemical analysis Vol I & II (6th Ed.)
14) S. N. Mahendru: Analysis of food products (Swan Publishers)

Paper No. - XV: Advanced Methods in Chemical Analysis
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to:

CO1: Understand the fluorescence and phosphorescence spectrophotometry. They will be able to explain the different types of luminescence, understand the theories behind fluorescence and phosphorescence, and discuss electronic transitions, solvatochromism, and solvation dynamics.

CO2: Adopt various kinetic methods of analysis. They will understand the theoretical basis behind kinetic techniques and be able to apply methods such as the Tangent Method, Fixed Time and Concentration Method, and Addition Method to determine the amount of substances in various samples.

CO3: Grasp the photoelectron spectroscopy and X-ray spectroscopy techniques. They will understand the basic principles of photoelectric effects, photoionization processes, Koopman's theorem, and the interpretation of photoelectron spectra.

CO4: Apply their knowledge of spectroscopic techniques to practical applications in analytical chemistry and research. They will understand how fluorescence sensing, synchronous spectra, and fluorescent nanomaterials can be utilized for specific analytical purposes.

UNIT-I: Fluorescence and Phosphorescence Spectrophotometry **(15)**

Fluorimetry, types of luminescence, Instrumentations, theories of fluorescence and phosphorescence, electronic transition, structural factors, solvatochromism, solvation dynamics, faith of excited molecules, solvent effect on fluorescence, effect of intermolecular process, fluorescence anisotropy and time domain fluorescence life time measurements. Relation between concentration with fluorescence and phosphorescence intensity, fluorescence quenching mechanism, resonance energy transfer. Chemiluminescence, Fluorescence sensing, Synchronous spectrum, Fluorescent nanomaterials. Practical applications, examples and problems in analytical chemistry and research.

UNIT-II: Kinetic Methods of analysis **(15)**

Theoretical basis of kinetic methods of analysis, methods of determining amount of the substance,

Tangent Method, Fixed Time and Concentration method. Addition Method, Oxidation Reactions of H2O2 with thiosulphate, iodide and amino, Enzyme catalyzed reactions. Inhibitors and Activators.

UNIT–III: Photoelectron spectroscopy **(15)**

Basic principles, photoelectric effects, Photoionization process, Koopman's theorem, photoelectron spectra of simple molecules, ESCA, chemical shift, Auger electron spectroscopy – basic idea.

UNIT-IV: X-ray spectroscopy **(15)**

Introduction, X-Ray generation, Properties of X-radiation, X-Ray, Instrumentation, X-Ray Absorption, Fluorescence and Diffraction methods of analysis and their applications

Recommended Books:

1) Gary D Christian, Analytical chemistry 6th edition. John Willey and sons INC (2003) H.
2) Kaur, Instrumental Methods of Chemical Analysis. Pragati Prakashan, Meerut.
3) W H Willard, L L Merritt and J A Dean, Instrumental Methods of Analysis.
4) S. M. Khopkar, Basic Concepts in Analytical Chemistry.
5) D. Skoog and D. West, Principle of Instrumental Analysis. Holl Seamlers.
6) E. Berlin, Principles and Practice of X-Ray Spectrometric Analysis, Plenum, NewYork.
7) J. Winefordner, S. Schulman and T O Haver: Luminescence Spectrometry in
8) Analytical Chemistry. Wiely Interscience New York.
9) H. Mark and G Rachnitz, Kinetics in Analytical chemistry. Interscience NY.
10) Gary D Christian,Analytical chemistry 6th edition.John Willey and sons INC (2003)
11) Engineering chemistry, R Gopalan, G. S. Nagrajan.
12) Engineering chemistry B. K. Sharma

Elective Papers
Paper No. – XVI (A): Industrial Analytical Chemistry
Theory: 60hrs

Course Outcomes: After the completion of the course, the student will be able to

CO1: Apply the various spectrochemical methods used for analytical purposes. They will understand the principles behind electronic spectra and molecular structure, be familiar with near-infrared (NIR) spectrometry for non-destructive testing, and comprehend the use of FTIR spectrometry, fluorometry, and optical sensors

CO2: Analyze metals and alloys. They will understand the composition analysis of foundry materials, ferroalloys, special steels, and various alloys like bronze, brass, Alnico, and Nichrome.

CO3: Understand the soil fertility determination, analysis of inorganic constituents in plant materials, and the chemical analysis as a measure of soil fertility. Students will be able to analyze fertilizers for their nutrient content and quality.

CO4: Understand the analysis of explosive materials such as TNT, RDX, lead azide, and EDNA. Additionally, students will be proficient in analyzing conducting polymers, resins, rubber, luminescent paints, lubricants, and adhesives, utilizing appropriate analytical techniques and methods.

UNIT-I: Spectrochemical Methods of Analysis **(15)**

Introduction to spectrochemical methods. Electronic spectra and molecular structure, NIR spectrometry for nondestructive testing. Solvents for spectrometry ,FTIR spectrometer ,fluorometry, optical sensors. Analysis of ores –bauxites, dolomites, monazites. Analysis of Portland cement.

UNIT-II: Analysis of metals and alloys **(15)**

Foundry materials, ferroalloys, and special steels, slags, fluxes. Analysis of alloys ,bronze, brass, Alnico and Nichrom

UNIT-III: Analysis of soil and fertilizers **(15)**

Method of soil analysis, soil fertility its determination, determination of inorganic constituents of

plant materials, Chemical analysis as measure of soil fertility, analysis of fertilizers.

UNIT-IV: Analysis of Commercial materials **(15)**

Analysis of explosive materials, TNT, RDX, lead azide, EDNA (ethylene dinitramine).Analysis of conducting polymer, resins and rubber. Analysis of luminescent paints, Analysis of lubricants and adhesive.

Recommended Books:

1) Hillebrand Lhundel, Bright and Hoffiman, Applied Inorganic Analysis, John Wiley.
2) Snell and Biffen, Commercial Methods of Analysis.
3) P.G. Jeffery, Chemical Methods of Rock Analysis, Pergamon.
4) Buchel, Chemistry of Pesticides. J Wiley.
5) Rieche, Outlines of Industrial Organic Chemistry, ButterWorth.
6) F.A. Henglein, Chemical Technology, Pergamon.
7) Kent, Riegl's Industrial Chemistry, Rainhold.
8) Chopra and Kanwar, Analytical Agriculture Chemistry, Kalyani Publishers.
9) Aubert and Pintes, Trace Elements in Soils.
10) Bear, Chemistry of Soil.
11) Hauson, Plant Growth Regulators, Noyes.
12) P.G. Jeffery and D.J. Hatchinson, Chemical Methods of Rock Analysis.
13) F.J. Weleher, Standard Methods of Chemical Analysis, A Series of Volumes Robert and Krigeger Publishing Company.
14) I. M. Kolthoff and PJ Ewing, Treatise o Analytical Chemistry, A series of Volumes.
15) R.D. Reeves and R.R. Brooks, Trace element Analysis of Geological Materials, John Wiley & Sons NewDehli.
16) W.M. Johnson and J.A. Maxwell, Rock and Mineral Analysis, John Wiley and Sons, NewYork.
17) W. F. Hildebrand, G H C Landell and HA Brighot, Applied Inorganic Analysis, John Wiley 2nd Edition.
18) K. J. Das, Pesticide Analysis (MD).

Paper No. – XVI (B): Quality Assurance and Accreditation
Theory: 60hrs

UNIT-I: Quality Assurance **(15)**

Introduction to Quality Control and quality assurance: Concepts and significance. Quality control and statistical techniques: Quality control charts, the X-quality control chart, the R-quality control chart and its interpretation, spiked sample control charts, use of blind samples in quality control, use of proficiency evaluations in quality control. Calibration and maintenance of Instruments / Equipment: Instrument calibration – linear calibration curves, equipment calibration, frequency of calibration, calibration of common laboratory instrument and equipment (Analytical balances, volumetric glassware, ovens, furnaces, UV / Visible spectrophotometer, pH meter, conductivity meter, IR spectrophotometers, AAS, GC, HPLC etc.,). Maintenance of instruments and equipment

UNIT-II: Documentation for Quality Assurance: Raw Data **(15)**

Type of notebooks, control of notebook distribution and data entry. General Reagents and volumetric reagents. Sampling – sampling methods, sample labelling, sample login/register. Sample analysis, reporting, recording and personal training.Instrument calibration and maintenance. Analytical report, Personnel, training, records - professional personnel, technician personnel. Filing quality assurance documentation. Good laboratory practices and personnel, Quality Programme, Instrument and Organisation calibration, Customer Satisfaction.

UNIT-III: Documentation for Quality Assurance: Raw Data **(15)**

Computers and quality assurance: Sample handling. Data Acquisition. Quality control data and calculations. Computer generated analytical reports. Security considerations. Hardware and software. Establishing a Quality Assurance program: Management commitment. Define the quality assurance program. Writing standard operating procedures. Topics for standard operating procedures. Consolidating the programme.Monitoring the program – monitoring quality assurance data, reporting quality assurance problems. Writing the quality assurance manuals.

UNIT-IV Quality Accreditation **(15)**

Laboratory Accreditation: Need for laboratory accreditation. International aspects of laboratory accreditation and in India. Criteria for laboratory accreditation. Benefits of laboratory accreditation,

Evolution and significance of Quality Management, Background to ISO 9000, comparison between ISO-9001, ISO-9002 & ISO-9003., ISO 9000-2000 series of standards on quality managamentsystem,- evolution of series of standards, introduction to ISO organization, Registration/ certification- benefits of QMS certification. Structure of ISO 9000-2000 family of standards. Advantages of ISO 9000-2000. Requirements of ISO 9001-2000 QMS and applications, Steps for effective implementations. Significance of ISO - 9001, 9002, 9003 & 9004. Requirements of ISO9000/ IS14001. Concepts of OHSMS (BS 8800) Quality Managament Principles in QMS, QMS documentation, Quality Manual, Quality policy, conformities and Nonconformities

Recommended Books:

1) Handbook of Quality Assurance for the analytical chemistry laboratory, James P. Dux, Van Nostrand Reinhold, New York, 1986.
2) Applying ISO-9000 Quality Management Systems, International Trade Centre Publishing, UNCTAD/WTO. Geneva, Switzerland, Indian Edition Printed by D.L.Shah Trust.
3) How to practice GLP, PP Sharma, Vandana Publications, 2000, New Delhi
4) Training manuals on ISO 9000 / 2000 PQM, Girdhar J Gyani, Raj Publishing House, 2001
5) Quality Assurance in Analytical Chemistry, B.W. Wenclawiak, Springer, India, 2004.

11. Determination of critical micelle concentration of given surfactants conductometrically
12. Estimation of acetyl salicylic acid in the given aspirin tablet by titrating against 0.1N alcoholic KOH potentiometrically.
13. To determine the acid base dissociation constant and isoelectric point of amino acid pH metrically
14. (Any other experiments may be added when required.)

(At least 6 major and 6 minor experiments should be carried out. More time should be given to project work)

B) Project:

Projects on contemporary issues of societal significance which should include literature survey, synthesis, reaction mechanism and kinetics, analysis of air, water and soil samples, solid state materials, energy generation and storage materials, nanochemistry, green chemistry, organic materials, organo-metallic, bioinorganic materials, novel materials etc. The Project/Review work (50 Marks) will be examined jointly by internal and external examiners at the time of practical examination.

(Any other experiments may be added when required.)

Study tour is compulsory for M. Sc. Part - II Students to visit Chemical Industries in India

CO-ORDINATOR
M.Sc.(ORGANIC & INORGANIC CHEMISTRY)
VIVEKANAND COLLEGE, KOLHAPUR
(EMPOWERED AUTONOMOUS)

VIVEKANAND COLLEGE KOLHAPUR (Empowered Autonomous) ESTD JUNE 1964

HEAD
DEPARTMENT OF CHEMISTRY
VIVEKANAND COLLEGE, KOLHAPUR
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