Rayat Shikshan Sanstha's Sadguru Gadage Maharaj College, Karad (An Autonomous College) SYLLABUS Semester – III and IV M. Sc. II (Analytical Chemistry) Implemented from Academic Year: 2023-2024

GENERAL OBJECTIVES OF THE COURSE:

- 1. To educate and prepare post graduate students from rural and urban area who will get employment on large scale in academic institutes, R & D and Quality control laboratories of Indian chemical/pharmaceutical industries as well as multinational and forensic Laboratories.
- 2. To provide students with broad theoretical and applied background in all specialization of Chemistry with emphasis on qualitative and quantitative technique.
- 3. To provide broad common frame work of syllabus to expose our young graduates to the recent and applied knowledge of interdisciplinary branches of chemistry involving applied organic, inorganic, physical, analytical, industrial, pharmaceutical, polymer, nano science & technology.
- 4. To conduct lesser written tests and to encourage on non-written tests.
- 5. To focus on encouraging students to conduct various academic activities like midterm tests, online tests, open book tests, tutorial, surprise test, oral, seminar, assignments and seminar presentation.

Learning Objectives:

- 1. A graduate with a Master's degree in Chemistry has in-depth and detailed functional knowledge of the fundamental theoretical concepts and experimental methods of chemistry.
- 2. The graduate has expert knowledge of a well-defined area of research within chemistry. The graduate has specific skills in planning and conducting advanced chemical experiments and applying structural-chemical characterization techniques. Skilled in examining specific phenomena theoretically and/or experimentally, the graduate is able to contribute to the generation of new scientific insights or to the innovation of new applications of chemical research.

Rayat Shikshan Sanstha's Sadguru Gadage Maharaj College, Karad (Autonomous) Department of Chemistry M.Sc. Analytical Chemistry: Course Structure-2023

Paper Code	Title of Paper	Evaluation Scheme (Marks)			Credits
i uper code		CCE	SEE	Total	Cicuits
M.Sc. I Analytical Semester- I					
A22-101	Inorganic Chemistry	20	80	100	4
A22-102	Organic Chemistry	20	80	100	4
A22-103	Physical Chemistry	20	80	100	4
A22-104	Analytical Chemistry	20	80	100	4
AP22-105	Chemistry Practical - I	-	100	100	4
AP22-106	Chemistry Practical - II	_	100	100	4
Non-CGPA	Communicative English-I	_	50	50	-
AAEC-I					
	Total	80	520	600	24
M.Sc. I Analytical Semester - II					
A22-201	Inorganic Chemistry	20	80	100	4
A22-202	Organic Chemistry	20	80	100	4
A22-203	Physical Chemistry	20	80	100	4
A22-204	Analytical Chemistry	20	80	100	4
AP22-205	Chemistry Practical – III	-	100	100	4
AP22-206	Chemistry Practical - IV	-	100	100	4
Non-CGPA	Fundamentals of Information Technology-I	-	50	50	-
ASEC-I					
	80	520	600	24	
M.Sc. II Analytical Semester- III					
A22-301	Principles of Analytical Chemistry	20	80	100	4
A22-302	Advanced Analytical Techniques	20	80	100	4
A22-303	Organo Analytical Techniques	20	80	100	4
A22-304	Electro Analytical Techniques	20	80	100	4
AP22-305	Chemistry Practical-V	-	100	100	4
AP22-306	Chemistry Practical-VI	-	100	100	4
Non-CGPA	Communicative English-II		50	50	-
AAEC-II					
	Total	80	520	600	24
WI.Sc. II Analytical Semester- IV					
A22-401	Techniques in Forensic Science and	20	80	100	4
	Microbiological Analysis	20		100	
A22-402	Environmental Chemical Analysis and Control	20	80	100	4
A22-403	Modern Separation Methods in Analysis	20	80	100	4
A22-404	Quality Assurance and Quality Control	20	80	100	4
AP22-405	Chemistry Practical-VII	-	100	100	4
AP22-406	Chemistry Practical-VIII	-	100	100	4
Non-CGPA ASEC-II	Fundamentals of Information Technology-II	-	50	50	-
AGE-I	Generic Elective		50	50	-
	Total	80	520	600	24
Grand Total		320	2080	2400	96

- The semester examination will be conducted at the end of each term (both theory and practical examination)
- Theory paper will be of 80 marks each and 20 marks for internal evaluation test conducted in the mid of the term. Two practicals will be of 100 marks each.
- Question papers will be set in the view of the entire syllabus and preferably covering each unit of the syllabus.

Laboratory Safety Equipment's:

Part: I Personal Precautions:

- 1. All persons must wear safety Goggles at all times.
- 2. Must wear Lab Aprons/Lab Jacket and proper shoes.
- 3. Except in emergency, over hurried activities are forbidden.
- 4. Fume cupboard must be used whenever necessary.
- 5. Eating, Drinking and Smoking in the laboratories strictly forbidden.

Part: II: Use of Safety and Emergency equipment:

- 1. First aid Kits
- 2. Sand bucket
- 3. Fire extinguishers (dry chemical and carbon dioxide extinguishers)
- 4. Chemical Storage cabinet with proper ventilation
- 5. Material Safety Datasheets.
- 6. Management of Local exhaust systems and fume hoods.
- 7. Sign in register if using instruments

M. Sc. II (Semester III)

A22-301 Principles of Analytical Chemistry (4 Credits)

Course objectives: Student should

1. To provide knowledge of basic principles of Analytical Chemistry.

- 2. To give idea about chemistry of Volumetric and Gravimetric Analysis.
- 3. To provide basic knowledge of Complexometric Equilibria
- 4. To give idea about chemistry of solvent extraction.

Unit I: Acid Base Equilibrium and Buffer Solution. (15 L)

Acid-base theories, Definition of pH and pH scale (Sorenson and operational definitions) and its significance, Hammett acidity function, pH at elevated temperatures, pH for aqueous solutions of very weak acid and base, pH for salts of weak acid and weak bases, polyprotic acids. Buffer solutions, buffer capacity, applications of buffers, Physiological buffers, buffers for biological and clinical measurements.

Unit II: Theory of Volumetric and Gravimetric Analysis. (15 L)

Introduction, Titrimetric analysis, classifications of reactions in titrimetric analysis, standard solutions, preparation of standard solutions, primary and secondary standards, Indicators, theory of indicators, Acid–base titrations in non-aqueous media. Gravimetric Analysis, Impurities in precipitates, Gravimetric calculations, precipitation equilibria (Solubility product, common ion effect, stoichiometry), organic precipitation.

Unit III: Complexometric Equilibria

Introduction, Titration curves, Types of EDTA titrations, Methods of End Point Detection(a)Indicators (b) Instrumental methods of End point detection

(Spectrophotometric, Amperometric, Potentiometric, High frequency titrator), Types of Complexometric Titrations (a) Direct Titration (b) Back Titration (c) Replacement titration (d) Indirect Titration (e) Applications of Complexometric Titrations

Unit IV: Solvent Extraction

Transition metal ions using ion exchanges. Basic principles, Classification of Solvents extraction systems, Extraction equilibria, Factors affecting extraction process, application of β - diketones, δ - Hydroxy quinoline, Di-thiocarbamates , Xanthates, Thiols, Separation of non-metals and metals.

Course Outcomes:

After completion of the units, students are able to:

1.Student will understand Acid base theory and all about buffer solution

2. Student will know Titrimetric analysis, Gravimetric Analysis,

Gravimetriccalculations

3.Student will understand types of EDTA titrations and types of Complexometric Titrations

4. Student will know Basic principles, Classification of Solvents extraction systems

Reference books:

1. D. A. Skoog and D. M. West, Fundamental of Analytical Chemistry, 7th Edition (1996), Saunders College Publishing, Philadelphia, Holt, London.

2. R. L. Pecsok, L. D. Shields, T. Cairns and L.C. McWilliam, Modern Methods of Chemical Analysis, (1976), John Wiley & Sons, New York.

3. D. A. Skoog, Principles of Instrumental Analysis, 5th Edition (1998), Saunders College of Publishing, Philadelphia, London.

4. H. A. Strobel, Chemical Instrumentation: A Schematic Approach, 2nd Edition (1973), Addison Wesley, Reading, Mass.

5. Analytical chemistry, G. D. Christian, Sixth Edition, Wiley publications

A22-302: Advanced Analytical Techniques.

Course objective: Student should

- 1. Study of Mass Spectrometry
- 2. Study of Advanced Instrumentation Techniques
- 3. To provide knowledge of Supercritical fluid chromatography
- 4. To give idea about Radio-analytical Chemistry

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

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 tector, 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 FTMS, and detectors

Unit II Advanced Instrumentation Techniques (15L)

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-III: Supercritical fluid chromatography (15L)

Introduction, SFC Advantages, Supercritical Fluids, Instrumentation, Supercritical Mobile Phase, Injectors, Ovens and Pumps, Columns, Detectors, SFC for the separation of polymers and of pesticides.

UNIT-IV: Radio-analytical Chemistry (15L)

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.

Course Outcomes:

After completion of the units, students are able to:

1. Student will understand Instrumentation, principles, history, concept of ion free path, classification of mass spectrometry

2. Student will know Introduction, principle, instrumentation, applications (SEM), (TEM), (EDS), (EDAX), (STM), (AFM).

3. Student will understand Instrumentation of Supercritical fluid chromatography.

4. Student will know Activation analysis, Sub-stoichiometric determination of traces of metals.

Reference books:

1. D. A. Skoog and D. M. West, Fundamental of Analytical Chemistry, International Edition, 7th Edition (1996), Saunders College Publishing, Philadelphia, Holt, London.

2. R. L. Pecsok, L.D. Shields, T. Cairns and L.C. McWilliam, Modern Methods of Chemical Analysis, 2nd (1976), John Wiley & Sons, New York.

3. L. R. Shyder and C. H. Harvath, An introduction to separation science, Wiley Interscience.

4. H. H. Willard; L. L. Merit; J. A. Dean & F. A. Settle, Instrumental Methods of Analysis(CBS).

A22-303: Organo-Analytical Techniques (4 Credits)

Course objective: Student should

- 1. To provide knowledge of Hyphenated Techniques
- 2. Students should know the Pharmaceutical Analysis
- 3. To give idea about Analysis of Dyes and paints
- 4. To provide knowledge of Analysis of oils, fats and Soaps & Detergents

Unit–I: Hyphenated Techniques

Concept of hyphenation, need for hyphenation, possible hyphenations. Interfacing devices and applications of GC- MS, ICP -MS, GC - IR, Tandem Mass Spectrometry, LC-MS: HPLC-MS, CE-MS.

Unit II: Pharmaceutical Analysis

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

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(15L)

antihistamines, sulfa drugs, barbiturates, etc. using non-aqueous titrations, sodium nitrite titrations, differential UV methods, colorimetric and fluorimetric methods of analysis.

Unit III: Analysis of Dyes and paints (15L)

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, flashpoint of paints. Practical applications and examples in analytical chemistry and research.

UNIT – IV: Analysis of oils, fats and Soaps & Detergents (15L)

Introduction to natural fats and oils, Analysis of oils and fats: Softening point, Congent point, Titre point, cloud point, Iodine, Saponification, acid, hydroxyl, R-M and Polenske value, Elaiden test.

Introduction to soaps, analysis of soap (saponifiable, unsaponifiable) and for unsaponified matter in soaps, Estimation of free alkali and phenol in soap. Classification of detergents (in Brief): Analysis of active ingredients from detergents (methylene blue and Hyamine-1622method); Estimation of CMC, Chlorides, total phosphates etc.

Course Outcomes:

After completion of the units, students are able to:

1.Student will be able to describe advanced techniques of analysis, Mass spectrometry. Students will be able to describe introduction to drugs, their classification, sources of impurities in pharmaceutical raw materials

2. Student will be able to describe composition of paint and practical applications.

3. Student will know Introduction to natural fats and oils, introduction to soaps, analysis of soap.

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 fregrance 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)

A22-304: Electro Analytical Techniques in Chemical Analysis (4 Credits)

Course objective: Student should

- 1. study of Voltammetry Techniques
- 2. Students should know the Ion selective electrodes & Electrochemical sensors
- 3. To give idea about Particle Size Analysis
- 4. To provide knowledge of Electrophoresis

UNIT-I: Voltammetry Techniques

Introduction, Principle, excitation signals in voltammetry, basic instrumentation based on operational amplifiers, voltammetric electrodes Cyclic Voltammetry: Instrumentation, Determination of analytes using cyclic voltammetry, 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: Ion selective electrodes & Electrochemical sensors: (15L) 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, ultra-micro electrodes and applications.

UNIT –III: Particle Size Analysis

(15L)

(15L)

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,

Photo sedimentation: 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:

(15L)

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 electrokinetic capillary chromatography, instrumentation, detection and applications and Applications, Numericals.

Course Outcomes:

After completion of the units, students are able to:

1. Student will be able to describe instrumentation, applications, cyclic voltammetry, Pulse voltammetry, Normal Pulse Voltammetry, Differential pulse voltammetry

2. Students will be able to describe types and construction of electrodes

3.Student will be able to describe Low angle LASER light scattering, Dynamic Light Scattering.

4. Student will know Paper electrophoresis, Techniques of electrophoresis.

Reference books:

1. R.D. Braum, Introduction to Instrumental Analysis.

2. Willard, Deritt, Dean and Settle, Instrumental methods of Analysis.

3. F. J. Welcher, standard Methods of chemical Analysis Vol.3, Part A & B.

4. G.W. Ewing, Instrumental Methods of Analysis 4th and 5th editions.

- 5. Chatwal and Anand, Instrumental Methods of Analysis.
- 6. Bassett, Denney-Jeffer and Mendham, Vogel's Textbook of Quantitative Inorganic Analysis.
- 7. Electro-analytical chemistry, edited by H.W. Nurnberg.
- 8. Kortum and Bockris, A Textbook of Electrochemistry.
- 9. D.A. Maclines, Principles of Electrochemistry.
- 10. Stulic, Ion selective electrodes (John Wiley).

AP22-305 and AP22-306: Analytical Chemistry Practical Course

Learning objectives: Students should

- 1) Learn analysis of ores and alloys
- 2) Study the estimations of pharmaceutical tablets, food samples.

3)Study the analysis of iodized salt, copper fungicide, vitamin-C in juices and squashes, ethambutol

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. To study the complex formation between Fe(III) and salicylic acid and determine the stability constants of the complex by Job's variation method.

11. To determine the equivalence conductance and dissociation constant using Kolhaursch Law at infinite dilution independent of ionic mobility of weak electrolyte.

12. Moisture analysis from drug/food sample by Karl-Fischer titration method.

(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 pKa of given dibasic acid pH-metrically.

19. Determination of relative strength of acetic acid, chloroacetic acid and trichloro acetic acid by conductometrically.

20. Determination of Cd and Zn from Zn-Cd mixture by polarography

Learning outcome:

After completion of experiments students are able to

- 1) Describe analysis of ores and alloys
- 2) Describe the estimations of pharmaceutical tablets, food samples.
- 3) Describe the analysis of iodized salt, copper fungicide, vitamin-C in juices

and squashes, ethambutol

References:

- 1) Handbook of Quantitative and Qualitative Analysis- H. T. Clarke
- 2) A Textbook of Practical Organic Chemistry A. I. Vogel
- 3) Findlay's Practical Chemistry Revised by J.A. Kitchner (Vedition)
- 4) Text Book of Quantitative inorganic analysis : A.I. Vogel.
- 5) Systematic Experimental Physical Chemistry :S.W. Rajbhoj andT.K. Chondhekar

M. Sc. II (Semester IV)

A22-401: Techniques in Forensic Science and Microbiological Analysis.

(4 Credits)

Course objective: Student should

1. To provide knowledge of Analysis of Body fluid and Clinical analysis Body fluid analysis

- 2. Students should know the Human Nutrition & Analysis of vitamins
- 3. To give idea about Pesticides Analysis
- 4. To provide knowledge of Forensic Analysis

Unit I: Analysis of Body fluid and Clinical analysis(15L)Body fluid analysis

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, cholesterol, urea, hemoglobin and bilirubin Urine- urea, uric acid, creatinine, calcium, phosphate, sodium, potassium and chloride.

Clinical Analysis

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, analytical methods to the constituents of physiological fluids (blood, urine and serum). Blood- Estimation of glucose, cholesterol, urea, hemoglobin and bilirubin, Urine- urea, uric acid, creatinine, calcium, phosphate, sodium, potassium and chloride.

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.

Course Outcomes:

After completion of the units, students are able to:

1. Student will be able to describe Body fluid analysis, Clinical Analysis

Unit II: Human Nutrition & Analysis of vitamins

a. Carbohydrates- Definition, functions and Analysis of total carbohydrates by Anthrone method, starch using Anthrone reagent, pectin by gravimetric method, and crude fibres.

b. Proteins- Definition, functions and analysis protein by Kjeldhal method and Lowry method, total free amino acids, methionine in food grain.

c. Vitamins- Definition, functions and analysis of Retinol, Vitamin D₃, Vitamin E, Vitamin B₁, Vitamin B₂, Vitamin B₆, Nicotinic acid, Niacin and Vitamin C.

d. Lipids- Definition, functions and analysis of free fatty acids, saponification value, iodine value and peroxide value.

Unit III: Pesticides Analysis

Introduction, classification of pesticides, sampling, sample pretreatment and processing, analysis of 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.

Special features of forensic analysis, sampling, sample storage, sample

dissolution, classification of poisons, lethal dose, significance of LD-50 and LC-

Unit IV: Forensic Analysis

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(15L)

2. Student will know Definition, functions and Analysis of Carbohydrates, Proteins, Vitamins, Lipids

3. Student will be able to describe Introduction, classification of pesticides

4. Student will know Special features of forensic analysis, sampling, Estimation of poisonous materials such as lead, mercury and arsenic in biological samples and Practical application.

Reference books:

- 1. S. R. Junk and H. M. Pancoast: Hand book of sugars(AVI)
- 2. B. Bilot and B. V. Well: Perfumery 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. Kramer Twigg: Quality control for food industry (AVI)
- 9. G. F. Longonan: the analysis of detergents and detergent products (JW)

10. A. Davidson & B. M. Milwidsky: Synthetic detergents (Book center, Mumbai)

- 11. M. Ash and L. Ash: A formulary of cosmetic preparations. (G. Goodwin)
- 12. Kurl Bauer, Dorothea Gadhe, 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)

A22-402: Environmental chemical analysis and control(4 Credits)Course objective: Student should

- 1. Students should know Sampling in Chemical Analysis
- 2. To give idea about Air and Water Pollutant Analysis
- 3. Students should know the Organic Pollutants and Their Analysis
- 4. To give idea about Removal of Heavy toxic metals

Unit I: Sampling in Environmental Chemical Analysis(15L)

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: Air and Water Pollutant Analysis

Chemistry of Air pollutants, characterization. source, methods of analysis of air pollutants; CO, CO₂, NOX, NH₃, H₂S, SO₂ 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 odor and its measurements.

Unit III: Organic Pollutants and Their Analysis (15L)

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

Unit IV: Removal of Heavy toxic metals(15L)

Cr, Hg, Pb, Cd, As, analytical methods of determination of small amount of

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metal pollutants, copper recovery, treatments of waste to remove heavy metals, recovery techniques.

Course Outcomes:

After completion of the units, students are able to:

- 1. Student will be able to describe Chemistry of Air pollutants, Chemical oxygendemand (COD) Biochemical oxygen demand (BOD)
- 2. Student will know Analysis of organochlorine pesticides
- 3. Student will be able to describe Introduction, classification of pesticides.

4. Student will know Laboratory Accreditation, introduction to ISO organization

Reference books:

1. A. K. De: Standard Methods of Waste and Waste water analysis.

2. S. M. Khopkar, Environmental Chemistry; Environmental pollution analysis.

3. M. S. Creos and Morr, Environmental Chemical Analysis, American publication (1988)

4. A. K. De, Environmental Chemistry, New Age International publishers.

5.Moghe, 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)
- 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.

A22-403: Modern Separation Methods in Analysis Course objective:

- 1. To provide knowledge of separation techniques.
- 2. Students should know chromatographic techniques in analytical chemistry
- 3. To provide knowledge of use of polymers in separation techniques.

UNIT-I: Advanced Gas Chromatographic Techniques

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

High Performance Liquid Chromatography (HPLC) and Ultra Performance Liquid Chromatography (UPLC)-Principle, instrumentation, mobile phase, Stationary support in HPLC, detectors and applications. Liquid Chromatography-Mass Spectrometry interface, instrumentation, advantages and applications. Practical applications and examples in analytical chemistry and research.

UNIT-III: Modern Extraction & Separation Techniques (15L) Modern extraction and separation techniques

Introduction, Theoretical aspects of extraction chromatography, solvent extraction and extraction chromatography with chelating ligands, extraction 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

(15L)

(15L)

(4 Credits)

chromatography with tributyl phosphate and other applications. Practical applications and examples in analytical chemistry and research.

UNIT-IV: Analysis of Polymers

(15L)

Molecular Weight of polymers- Arithmetic, weight average and number average molecular weights of polymers, Determination of molecular weight of polymers, End-Group Analysis, Cryoscopy, Light scattering, Viscosity, and gel permeation chromatographic methods, Thermal Transition in Polymers, T_g and T_m and their relation, DSC, TGA, Spectral methods of Analysis.

Reference books:

1. D. A. Skoog and D. M. West, Fundamental of Analytical Chemistry, International Edition, 7th Edition (1996), Saunders College Publishing, Philadelphia, Holt, London.

2. R. L. Pecsok, L.D. Shields, T. Cairns and L.C. McWilliam, Modern Methods of Chemical Analysis, 2nd(1976), John Wiley & Sons, New York.

3. L. R. Shyder and C. H. Harvath, An introduction to separation science, Wiley Interscience.

4. H. H. Willard; L. L. Merit; J. A. Dean & F. A. Settle, Instrumental Methods of Analysis(CBS).

A22-404: Quality Assurances and Quality control.

(4 Credits)

Course objective:

- 1. To provide knowledge of Quality Assurance
- 2. To provide knowledge of Quality Accreditation
- 3. Students should know the Documentation for Quality Assurance: Raw Data
- 4. To give idea about Computers and quality assurance
- 5. To provide knowledge of Development and validation of Analytical Method

UNIT-I: Quality Assurance

(15L)

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: Quality Accreditation

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 management system, 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 Management Principles in QMS, QMS documentation, Quality Manual, Quality policy, conformities and Nonconformities.

UNIT-III: Development of Analytical Method (15L)

Theory and factors affecting resolution – a reminder of the importance of resolution, separation factor (selectivity), retention factor (capacity factor) and column efficiency). Selecting the HPLC separation mode (reversed-phase, normal-phase *etc.*) Selecting the most appropriate detector Gradient/isocratic operation, selecting the column for analysis, Selecting and optimizing the mobile phase, the effect of pH, considering pKa of the analyte Requirements for a stability-indicating analytical method, Anticipation of likely degradation products, From experience with compound, From forced degradation (stress testing) of drug substance, as per ICH guidance, note findings of stress-testing industry comparison, Are degradation products likely to be enantiomers or distereoisomers, Calculation of mass balance and its significance.

UNIT- IV: Validation of Analytical Method:

Introduction to ICH guidelines: ICH Q2(R1), A detailed discussion on the parameters to be validated, Specificity: peak purity determination (Diode array and MS detectors), Linearity, Range, Accuracy, Precision, Detection Limit, Quantitation Limit, Robustness. Extent of validation: how much work at each phase of development, Acceptance criteria, Validation procedures and protocols, Dealing with validation failures.

Course Outcomes:

After completion of the units, students are able to:

- 1. Describe Quality Control and quality assurance
- 2. Describe quality accreditations in details.

3. Describe quality assurance, quality assurance program, Calculation of mass balance and its significance

4. Know an Analytical method development, Validation of methods

Reference books:

1. D. A. Skoog, D. M. West and F. J. Holler, Fundamentals of Analytical Chemistry, 2nd Ed., Saunders College Publishing, 1991.

2. R. A. Day and A. L. Underwood, Quantitative Analysis, 6th Ed., Prentice-Hall of India Pvt. Ltd., 1993.

- 3. Gas Chromatography, Open Book Learning Series
- 4. Larry Hargis, Analytical Chemistry. Principles and techniques
- 5. Encyclopedia of Analytical Chemistry, Vol.

AP22-405 and AP22-406: Analytical Chemistry Practical Course

Learning Objective: students should

1) Learn analysis by using different instruments.

- 2) Learn Analysis of milk, pesticides, insecticides, cements, soil etc.
- 3) Study estimations of salicylic acid, copper, urea etc.

Major

1. Cement analysis.

2. Analysis of Chrome steel alloy for Cr and Ni content.

3. Analysis of bauxite ore to estimate the amount of silica, aluminium and iron.

4. Estimation of salicylic acid and zinc oxide from medicated powder.

5. Determination of saponification value and iodine value of oil.

- 6. Estimation of amount of copper (II) with EDTA spectrophotometrically.
- 7. Simultaneous spectrophotometric determination of Cr and Mn.
- 8. Analysis of milk.

9. Analysis of some common pesticides, insecticides, plastics and detergents.

10. Estimation of Urea, Uric acid and creatinine in Urine.

11. Estimation of blood sugar, calcium and total nitrogen and non-protein nitrogen in blood.

12. Studies on the effect of substituent at ortho position of benzoic acid on its equilibrium constant pH metrically.

13. Agricultural analysis of soil sample, animal feeds, soil micronutrients, milk powder for Ca, Fe and P content.

Minor

- 1. Estimation of Fe from soil sample
- 2. Analysis of Na and K from soil sample
- 3. Determination of chemical oxygen demand of water sample (dye solution)
- 4. Estimation of lactose from milk sample
- 5. Determination of flash point of oil/fuel

6. To estimate the amount of glycine from amino acid

7. To determine the amount of alkali content of antacid tablet titrimetrically

- 8. Determination of dissociation constant of weak acid pH-metrically.
- 9. Estimation of Zn in the given solution fluorimetrically.
- 10. Determination of pK of tribasic acid, by potentiometry.
- 11. Determination of critical micelle concentration of given surfactants conductometrically.

12. Estimation of acetyl salicylic acid in the given aspirin tablet by titrating against 0.1Nalcoholic KOH potentiometrically.

13. To determine the acid base dissociation constant and isoelectric point of amino acid pH metrically

Any other experiments may be added when/if required

N B. 1. At least 6 major and 6 minor experiments should be carried out.2. More time should be given to project work

Project: Lab Project in Parent Institute

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, nano-chemistry, 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.

OR

Internship, Industrial Training:

Students should complete their internship /industrial training / research project work in well reputed organizations like IIT, NIT, IISER, NCL, ICT, Universities, Chemical Industries and Pharmaceutical Industries etc. The minimum period of their internship is 30 days. Students should submit their project report in details with certificate is mandatory.

Learning Outcome: After completion of experiments, students are able to

- 1) Describe analysis by using different instruments.
- 2) Describe Analysis of milk, pesticides, insecticides, cements, soil etc.
- 3) Describe estimations of salicylic acid, copper, urea etc.

References:

- 1) Handbook of Quantitative and Qualitative Analysis- H. T. Clarke
- 2) A Textbook of Practical Organic Chemistry A. I. Vogel
- 3) Findlay's Practical Chemistry Revised by J.A. Kitchner (Vedition)
- 4) Text Book of Quantitative inorganic analysis : A.I. Vogel.
- 5) Systematic Experimental Physical Chemistry :S. W. Rajbhoj

andT. K. Chondhekar