

M Sc Semester III Analytical Chemistry

Semester III CHE2301AC

Sample preparation, Chemometrics and Classical Methods of Analysis -3 credits

Unit I – Sample preparation for Analysis

- *Selection of an analytical method:* Factors to be considered, possible causes of error.
- *Sampling and sample preparation:* Factors involved in effective sampling, designing a sampling plan, concept of sample size, quality of sample, sample registration and storage, methods of physical separations in sample preparation
- *Solid Phase Extraction-* Introduction, sorbents, matrix solid phase dispersion and applications of SPE in environmental and biological samples
- *Solvent extraction:* Crown ethers, cyclodextrins, cryptands and calixarenes and rotaxanes in solvent extraction, Synergistic extractions. Methods for improvement of selectivity and sensitivity of multiple extractions. Solvent impregnated resins., Accelerated solvent extraction, microdrop solvent extraction, solvent extraction using reverse micelles, cloud point extraction. Special applications of solvent extraction .

Unit II Chemometrics

- *Introduction to Chemometrics,* one-tailed and two-tailed tests, errors in significance testing, detection limits, ANOVA – one way, analysis of residuals.
- *Calibration and standardization:* single point and multiple point, external, internal standardization, method of standard addition. *Calibration curve:* correlation and regression, unweighted and weighted linear regression, blank corrections.
- *Experimental design:* randomization, two way analysis of variance, factorial design, Yates method, Optimization methods, introduction to treatment of multivariate data.
- *Statistical approach to biological samples:* Principles of experimental design, completely randomized design (CRD), randomized block design (RBD), analysis of variance for these designs.
- Signal to noise ratio and methods for its enhancement.

Unit III Classical Methods of analysis

- *Classical methods of analysis:* Concentration units, chemical equilibrium and its calculations.
- *Analytical methods based on mass measurement:* Principles, stoichiometric calculations, precautions needed in analysis.

- *Analytical methods based on volume measurement:* Principles, direct and indirect titrimetric methods, non aqueous titrations, titration curves, ways to minimize errors in analysis.

BOOKS RECOMMENDED

1. Analytical Chemistry, G.D. Christian, John Wiley & Sons. 6th Edition.2004
 2. Quality in the Analytical Chemistry Laboratory, E. Prichard, John Wiley & Sons, N.Y.
 3. Vogel's Textbook of Inorganic Quantitative Analysis; Pearson Education Ltd
 4. Quality Assurance in Analytical Chemistry W. Funk, V. Dammann, G. Donnevert VCH Weinheim (1995).
 5. Solvent Extraction of Metals, A.K. De, R.A. Chalmers and S.M. Khopkar, Van Nostrand Reinhold.
 6. Solvent Extraction Chemistry, T. Sekine and Y. Hasegawa, Marcel Dekker and Co. (1980).
 7. Solvent extraction principles and application to Process Metallurgy (Part I). G.M. Riterly and A.W. Ashbrook, Elsevier (1984).
 8. Solvent Extraction and Ion Exchange- J Marcus and A.S. Kertes, Wiley Interscience (1969).
 9. Supercritical fluid extraction – Larry Taylor-Wiley publishers- New York (1996).
 10. Chemical separations and Measurements Introduction to Separation Science, B.L. Karger, L.R. Snyder, C. Howath, John Wiley N.Y. (1973).
 11. Principles and Practice of Analytical Chemistry, F.W. Fifield, 5th Ed. (2000).
 12. Encyclopedia of Analytical Science Academic Press, New York, Vol. 1 to 10 Chief Editor Allen Town Shend.
 13. Principles of Analytical Chemistry M. Valcarcel, Springer,(2005).
 14. Quantitative Analysis, R.A. Day and A.Z. Underwood,. Prentice Hall. Inc.3rd Ed.
 15. Analytical Chemistry of macrocyclic and supramolecular compounds, S.M.Khopkar, Narosa Publishing house 2005
 16. Basic concepts of analytical chemistry, S.M.Khopkar, New Age International, 1998
 17. Statistics and chemometrics for Analytical chemistry, J.C.Miller and J.N.Miller, Prentice Hall
 18. Sample Preparation Techniques in Analytical Chemistry, Somenath Mithra, Wiley Interscience,2003
 19. Solid-phase extraction: principles, techniques, and applications, [Nigel J. K. Simpson](#),CRC, 2000
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M Sc Semester III **Analytical Chemistry**

CHE2302AC **Advanced Chromatographic Techniques -3 credits**

Unit I Chromatography - I

- High Performance Liquid Chromatography (HPLC): Introduction.
- Columns for HPLC: preparation of column materials and packings, maintenance of columns, micro and capillary columns. Advances in column technology.
- Isocratic and gradient programmers.
- Method development and validation: Choice of columns and solvents, elution strength of solvents, use of buffers and special agents in analysis.
- *Derivatisation in HPLC*: Post and Pre-column.
- Important applications, including separation of chiral compounds.
- Multidimensional HPLC

Unit II Chromatography - II

- *Detectors for HPLC*: Instrumentation and applications of absorption based detectors including diode array, RI, fluorescence, ELSD, Charged Aerosol detectors. Indirect detection.
- Fast HPLC, Preparative HPLC, Troubleshooting in HPLC. Advances in detection techniques.
- *High Performance Thin Layer Chromatography (HPTLC)*: Principles and instrumentation, HPTLC vs. TLC, densitometry and quantitation in HPTLC. HPTLC in fingerprinting and QC. Applications of HPTLC.

Unit III Chromatography - III

- *Gas Chromatography (GC)*: Introduction.
- *Columns for GC*: Packed and capillary column, column packings and stationary phases, chiral columns. Preparation and care of GC columns. Equivalent columns.
- Split and split-less injection systems, head space sampling, SPME, pyrolysis, Advances in detection techniques.
- *Introduction to Multidimensional GC* and Fast GC.
- Important applications of GC.
- *GC Method development and validation*: Choice of columns, polarity indices - Kovat's retention index, Mc Reynold's constants, resolution of enantiomers. Troubleshooting in GC.

BOOKS RECOMMENDED

- 1 Chromatography: Part A & B, E. Heftman, Elsevier Science Publishing. 5th Edition.
- 2 Chromatography today: C.F. Poole & S.K. Poole, Elsevier Science Publishing . (1991)
- 3 Instrumental Analysis , Y.H. Bauer, G.D. Christian, S.E. O'reilly, Allyn and Bacon Inc.
- 4 Introduction to Instrumental Analysis , R. D. Braun, McGraw-Hill International Editions.
- 5 Instrumental methods of analysis H.H. Willard, L.L. Merritt, J.N. Dean and F.A. Settle, I.B.H. Publishing House, New Delhi.
- 6 Chromatography: Concepts and contrasts J.M. Miller Jhon Wiley & sons Inc.
- 7 Practical High performance Liquid Chromatography; V.R. Meyer, John Wiley, New York

M Sc Semester III Analytical Chemistry

CHE2303AC Advanced Spectroscopy and Microscopy -3 credits

Unit I *Molecular spectroscopy*

- **NMR** : Recapitulation of basic concept , NMR instrumentation, FT-NMR, Pulse excitation, Pulse Sequences, Practical Considerations, Qualitative and quantitative analysis. Non protonic NMR spectra, Nuclear overhauser effect, Multiple resonance , 2- D NMR, COSY, HETCOR, TOCSY, NOESY , solid state NMR, Magnetic Imaging.
- *CD-ORD*, circular birefringence, circular dichroism, plain dispersion curves , single and multiple cotton effect curves, structural and stereochemical applications – axial haloketone rule, octant rule for ketones, Comparison of ORD and CD

Unit II *Atomic spectroscopy*

- *Atomic Absorption Spectroscopy* Principles- spectral line width, doppler and pressure broadening, Atomization process, Atomic line widths and radiation sources for AAS, temperature gradients, cells detectors, interferences, Background correction methods and modifications in instrumentations,
- *Atomic Emission Spectroscopy*: Atomic spectra, Population distribution with temperature, Sources, spark laser microprobe for atomic emission, Spectrometers, Merits, demerits, and applications

Unit III *Spectroscopic and Microscopic techniques*

- Introduction to ultrahigh vacuum;
- X-ray photon spectroscopy (XPS), Auger Electron Spectroscopy (AES), Ultraviolet photon spectroscopy (UPS) and Sum Frequency Generation Spectroscopy : basic principle, instrumentation, data interpretation and analysis,
- Time-of-flight secondary ion mass spectrometry (ToF-SIMS); basic principle, instrumentation, data interpretation and analysis,
- *Scanning electron microscope* (SEM) and *Atomic force microscope* (AFM): basic principle and instrumentation.
 - *Photoacoustic spectroscopy* :Theory, instrumentation, qualitative and quantitative analysis

BOOKS RECOMMENDED

1. Instrumental methods of analysis H.H. Willard, L.L. Merritt, J.N. Dean and F.A. Settle, I.B.H. Publishing House, New Delhi.
2. Analytical Chemistry, G.D. Christian, John Wiley & Sons. 6th Edition, 2004

3. Principles of Instrumental analysis, D.A. Skoog, F.J. Holler and T.A.Nieman., Saunders College Publishing. 5th Edition
 4. Instrumental Analysis , Y.H. Bauer, G.D. Christian, J.E.O' Reilly. Allyn and Bacon, Inc.
 5. Introduction to Instrumental Analysis , R. D. Braun, PharmaMed Press.
 6. Principles and Practice of Analytical Chemistry, F.W. Fifield, 2nd Ed. (1983).
 7. Encyclopedia of Analytical Science Academic Press, New York, Vol. 1 to 10 Chief Editor Allen Town Shend.
 8. Fundamentals of Molecular spectroscopy, C. Banwell and E. McCash, Tata McGraw-Hill International edition
 9. Introduction to Spectroscopy, D.Pavia, G. Lampman, G.Kriz Thomson Brooks Cole
 10. Modern Molecular Spectroscopy, H.S. Randhawa, Mcmillan India Ltd.
 11. Electronic Absorption Spectroscopy and related Techniques; D.N. Sathyanarayana, Universities Press.
 12. Principles of Analytical Chemistry M. Valcarcel, Springer,(2005).
 13. Quantitative Analysis, R.A. Day and A.Z. Underwood,. Prentice Hall. Inc.3rd Ed.
 14. Surface analysis: The Principal Techniques, John .Vickerman, Ian Gilmore, John Wiley and sons, 2009
 15. Surface analysis methods in materials science, By D. John O'Connor, Brett A. Sexton, Roger St. C. Smart, second edition springer,2003
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M Sc Semester III Analytical Chemistry

Practical Courses

Advanced Analytical chemistry practicals-1CHE2304AC, Advanced Analytical chemistry practicals-2CHE2305AC

(8 credits)

Separation techniques

Separation of Iron and Nickel ions from mixture by solvent extraction and their determination

Determination of Nickel using DMG by molten-naphthalene extraction

Determination of Nickel using DMG by solvent extraction

Determination of copper using diethyl dithiocarbamate by solvent extraction

Determination of iron using oxine by solvent extraction

Determination of SiO_2 , CuO and Fe in copper pyrite

Determination of Lead (Pb) and Tin (Sn) in solder

Separation and determination of Zinc and Magnesium on anion exchanger

Separation and determination of Zinc and Cadmium using ion exchanger

Determination of Cl^- and Br^- in a mixture by ion exchange method

Separation and determination of Cu and Fe in a given mixture

Separation and determination of Fe and U in a given mixture

Determination of ion exchange capacity of a given ion exchanger

Separation and determination of molecular weight of proteins by electrophoresis

Separation and determination of copper and cadmium by polarographically

Determination of phenol from soil sample by Soxhlet extraction

Determination of nitrogen content of given fertilizer sample by Kjeldahl method

Flue gas analysis by Orsat's apparatus

Water treatment

Determination of free chlorine and available chlorine in water

Determination of COD in a given water sample

Determination of phosphate in water spectrophotometrically in a given water sample.

Determination of ammonia in water spectrophotometrically

Determination of ammonia / phosphate in water by standard addition technique

Determination of dissolved oxygen in water

Determination of sulphate in water by turbidimetry

Determination of Cr in water sample using diphenyl carbazide

Determination of hardness of water

Determination of acidity and alkalinity of given water sample

Determination of nitrate and nitrite content of given water sample

Determination of Cu/Zn in water sample by dithizone

Determination of chloride in water sample

Pharmaceutical and food analysis

Determination of diclofenac in given tablet by non –aqueous titration

Determination of Mg^{+2} and Al^{+3} in digene tablet

Determination of ascorbic acid in vitamin C

Determination of friability and hardness of given tablet

Study the disintegration of a given tablet

Determination of protein content of a milk product by formalin method

Determination of protein content of a milk product spectrophotometrically by Lowry method

Determination of Ca in tablets or milk products

Determination of peroxide value of oil

Determination of iodine value of oil

Determination of moisture content in food/ pharmaceutical sample by Karl Fischer titration

Determination of thiamine / quinine fluorimetrically

Determination of Chloride/ bromide fluorimetrically by quenching technique

Determination of enzyme activity spectrophotometrically

Determination of Li in tablets by flame photometry

Miscellaneous

Determination of pKa of indicator spectrophotometrically

Biamperometric titration of Fe(II)

Plateau characteristic of GM Counter

Determination of the dead time of a GM counter

Statistical aspects of radioactivity Measurements

M Sc Semester III Analytical Chemistry

CHE2301AE

Environmental Chemistry and Green Chemistry- 2 credits

Unit-I Environmental Chemistry

- *Environmental Chemistry of water* Properties of Water, Major aquatic chemical processes
- *Water Pollution*: Types, sources and consequences of water pollution. sewage and waste water treatment and recycling
- Environmental Impact Assessment. Environmental Auditing. Carbon credits
- *Soil Pollution*: Physical and chemical properties of soil ,soil micro-organisms and their functions, Different kinds of synthetic fertilizer (N, P and K) and pesticides-their interactions with different components of soil. their toxicity and pollution. Industrial effluents of different kinds, their interactions with soil components, problems of toxic heavy metals and pollution. Deterioration of soil due to mining activities .Soil Pollution Control

Unit II– Green Chemistry

- Green Chemistry: Definition, tools and principles of green chemistry, evaluation of starting materials and reaction types.
- solvent free systems, supercritical fluids, ionic liquids as catalysts and solvents
- Emerging Green Technologies: Photochemical reactions (advantages and challenges), examples, chemistry using microwaves, sonochemistry, electrochemical syntheses.
- Waste Minimization techniques: Concept of Recycling, recovery and reuse, Bioremediation of wastes and soils, phytoremediation of soils and sediments

BOOKS RECOMMENDED

1. Environmental Pollution analysis, S.M. Khopkar ,Wiley Eastern Ltd.,
 2. Environmental Chemistry , S.K. Banerji, Prentice Hall of India Pvt. Ltd., New Delhi
 3. Environmental Chemistry, Stanley Manahan, Lewis Publishers CRC Press, USA. , 6th Edition
 4. Identification and Analysis of Organic Pollutants in water L.H. Keith, Arber Science Publications.
 5. Standard methods for examination of water and waste water analysis, American Public Health Association, New York.
 6. Chemical treatment and water purification, F .J. Belan, Mir Publishing House, Moscow.
 7. Standard Methods for Examination of Water and waste water analysis American Public Health Association, N.Y.
 8. Water and water pollution (Hand Book) Ed., Seonard'L. Ciacere, Vol. (I to IV), Marcel Dekker Inc. N.Y. (1972).
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M Sc Semester III Analytical Chemistry

CHE2302AE

Mass spectrometry and hyphenated techniques- -2 credits

Unit-I *Advanced Mass Spectrometry-*

Field Ionization and Desorption Mass Spectrometry, Plasma Desorption Mass Spectrometry

Isotope Dilution Mass Spectrometry, Resonance Ionization Mass Spectrometry, Continuous Flow Fast Atom Mass Spectroscopy, Atmospheric Pressure Ionization Mass Spectroscopy

Unit-II *Hyphenated techniques*

Introduction, need for hyphenation, Advantages and problems involved in hyphenation, possible hyphenation. Interfacing devices and applications of GC-MS, GC-IR, MS-MS, HPLC-MS, ICP-MS.

Books Recommended:

1. GC/MS, A Practical User Guide : M. McMaster & C. McMaster Wiley - VCH 1998
2. Fundamentals of Analytical Chemistry”, D.A. Skoog, D.M. West, F.J. Holler, S.R.Crouch 8th, edn.
3. Instrumental Methods of Analysis H.H. Willard, L.L. Merritt Jr., J.A. Dean, F.A. Settle (CBS Publisher) 7th edn.

M Sc Semester III **Analytical Chemistry**

CHE2303AE **Food and Polymer Analysis – 2 credits**

Unit I *Food Analysis*

- Introduction, standards, food legislation and nutritional evaluation.
- General chemical methods: Sampling and sample preparation, moisture, ash and mineral matter, sodium chloride, titratable acidity, nitrogen and crude protein, fat, characterization of fat, crude fiber and dietary fiber, foreign bodies in food.
- Analysis of milk, flour, honey, edible oil and beverages.

Unit II *Polymer Analysis*

Polymer Characterization Techniques:

- molecular weight and mol. wt. distribution and end group analysis
- thermal and thermomechanical analysis (TGA, DTA/DSC, TMA, DMA)
- Gel Permeation Chromatography
- Viscosity
- Nuclear Magnetic Resonance spectroscopy (^1H -NMR, ^{13}C -NMR, multinuclear NMR, 2D NMR, monomer sequence, copolymer composition, branching, mol. wt. determination by NMR);
- Infrared spectroscopy
- analysis by microscopic techniques (optical, phase contrast, SEM, TEM); ESCA and other technique

Books Recommended

1. Analysis of Foods and Beverages, George Charalambous, Academic Press.
2. Chemical analysis of food and food products, H.B. Jacob, Van Nostrand Reinhold.
3. Introduction to food science and technology, G.F. Stewart and M.A. Amerine, Academic Press
4. The Chemical Analysis of food, Pearson
5. Indian Pharmacopoeia
6. Textbook of Polymer Science, Fred W. Billmeyer Jr., John Wiley & Sons Inc.
7. Polymer Characterization, E.Schröder, G.Müller, K.F.Amt, Hanser Publishers
8. Simple Methods for Identification of Plastics, D.Brown, Hanser Publishers

M Sc Semester III Analytical Chemistry

CHE2304AE Forensic Analysis -2 credits

Unit I-

- **History and Development of Forensic Science**, Definition of Forensic Science, Scope of Forensic Science, Need of Forensic Science, Basic Principles of Forensic Science, Tools and Techniques of Forensic Science International Perspective of Forensic Science
- **Forensic Chemistry**: Introduction, Types of cases which require chemical analysis, Limitations of forensic samples, conventional methods of chemical analysis, presumptive tests (colour/spot tests), Microcrystal tests, Elemental analysis (organic and inorganic).
- **Forensic examination of Body fluids**: 1) Blood: Identification (Preliminary and confirmatory tests), species of origin (Immunodiffusion and Immunoelectrophoresis), Individualization: Blood grouping, enzyme typing
2) Semen: Composition, functions and morphology of spermatozoa, Identification (Preliminary and confirmatory tests including Azoospermic semen stains), Individualization (Blood Grouping, seminal fluid isozymes typing)
3) Composition, functions and forensic significance of saliva, sweat, milk, urine, faecal matter, vaginal secretions and tests for their identification including the presence of blood group specific ABH substances.
- Physical Examination of blood stains
- Fiber Examination: Introduction, Classification, Fibre transfer and persistence. Fiber Recovery: At the scene, in the laboratory, contamination and its prevention. Fiber Identification and comparison: Microscopical Examination, Determination of optical properties, Refractive Index, Birefringence, Instrumental analysis, dye analysis by TLC, factors affecting the strength of fibre evidence.
- Hair examination: Hair structure, growth and replacement of hair.
Identification: Species of origin, variation in different major population groups, somatic origin.

Unit II

- **Forensic Toxicology**: Introduction, Role of the toxicologist, significance of toxicological findings, poisons, definition, classification on the basis of their origin, physiological action and chemical nature, poisons and poisoning in India
- Analysis of Narcotic Drugs and Psychotropic Substances: Job of forensic drug chemist, analysis of NDPS evidence by various procedures prescribed by U.N. Manual, DFS manual, spot tests, microcrystal tests, extraction methods, TLC, UV-Vis spectrophotometry, IR spectrophotometry, GC-HPLC, MS, GC-MS, NMR and XRD as exemplified by cocaine, cannabis, barbiturates, benzodiazepines, amphetamines, opiates and hallucinogens (LSD, psilocybine and mescaline).
- Toxicology of Alcohol, fate of alcohol in body, tests for alcohol, Detection and determination of ethanol, furfural, organic acids, aldehydes, chloral hydrate, methanol and ethylene glycol in liquors by color tests, TLC, GC, and GC-MS methods.

- Explosives: Classification, composition and characteristics of explosives, pyrotechnics, IEDs, explosion process and affects, types of hazards, effect of blast wave on structures, human etc, specific approach to scene of explosion, post blast residue collection, reconstruction of sequence of events, evaluation and assessment of scene of explosion, systematic examination of explosives and explosion residues in the laboratory using chemical and instrumental techniques and interpretation of results.
- Some real case analysis

Books Recommended

1. Curry : Analytical Methods in Human Toxicology, Part II,
 2. Casarett & Doll Toxicology : The Basic Science of poisons.
 3. Clark, E.G.C. : Isolation and identification of Drugs, VI and Vol. II,
 4. Curry, A.S. : Poison Detection in Human Organs, 1976.
 5. Curry, A.S. : Advances in Forensic Chemical Toxicology, 1972.
 6. Holfmann, F.G. : Handbook of Drug and Alcohol Abuse.
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M Sc Semester III Analytical Chemistry

CHE2305AE ***Advanced Analytical Techniques-2 credits***

Unit-1 Automation in Analysis

- *Automated Systems*- An overview, distinction between automatic and automated systems, advantages and disadvantages of automation, types of automated techniques.
- Non-discrete techniques, segmented flow methods and basic equipments, special techniques and devices, theoretical considerations and problems, applications. Single and multi channel auto analyzers, BUN analyzers, automatic glucose analyzer and ammonia in water analyzer, COD analyzer. Non-segmented flow methods.

Unit-2 Special topics in chromatographic techniques

- *Affinity chromatography*: Separation, mechanism, matrices, matrix activation, role of spacer groups, applications.
- *Size exclusion chromatography*: introduction, instrumentation, applications.
- *Ion Chromatography*: Principles of separation and detection, instrumentation, types of resins, detectors, applications.

• **BOOKS RECOMMENDED**

1. Principles of Instrumental Analysis, D.A. Skoog, F.J. Holler and T.A. Nieman 5th Edition (1998), Saunders College Publishing, Harcourt Brace & Company, U.S.A.
2. Automation in the laboratory By William Jeffrey Hurst, VCH, 1995
3. Ion Chromatography J.S. Fritz, D.T. Gjerde 3rd edition(2000) Wiley VCH
4. Chromatography: Part A & B, E. Heftman, Elsevier Science Publishing. 5th Edition.
5. Chromatography today: C.F. Poole & S.K. Poole, Elsevier Science Publishing . (1991)
6. Instrumental Analysis , Y.H. Bauer, G.D. Christian, S.E. O'reilly, Allyn and Bacon Inc.

M Sc Semester IV Analytical Chemistry

CHE2401AC

Spectral and Kinetic Methods of Analysis-3 credits

Unit-I ESR: Introduction: Similarities between ESR and NMR, g-values and factors affecting it, determination of g value, Hyperfine interaction, FT ESR, Electron Nuclear double resonance (ENDOR), Electron double resonance(ELDOR)

Unit-II Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy: Basic principle, instrumentation, data interpretation and analysis, attenuated total reflection , diffuse reflectance, and Polarization modulation-infrared reflection-adsorption spectroscopy.

Unit III Flow Injection and Kinetic Methods of Analysis:

- *Flow injection analysis-* Principle, types of dispersion, factors affecting dispersion, applications of small, medium and large dispersion, stopped flow methods, flow injection titrations.
- *Discrete methods-* Centrifugal fast scan analyzer, automatic multipurpose analyzer, automatic elemental analyzer, automated analyzer based on multilayer film principles, film structure, instrumentation applications
- *Kinetic methods of analysis:* Use of reaction rates in analysis. Factors affecting rate of reaction, methods of determination of order of reaction. Types of kinetic methods (differential, integral and fractional change method and multi component analysis). Instrumentation, quantitative applications of the enzyme catalyzed reactions, non enzyme catalyzed reactions and non catalytic reactions. Evaluation of chemical kinetic methods

• BOOKS RECOMMENDED

1. Instrumental methods of analysis H.H. Willard, L.L. Merritt, J.N. Dean and F.A. Settle, I.B.H. Publishing House, New Delhi.
2. Analytical Chemistry, G.D. Christian, John Wiley & Sons. 6th Edition, 2004
3. Principles of Instrumental analysis, D.A. Skoog, F.J. Holler and T.A. Nieman., Saunders College Publishing. 5th Edition
4. Instrumental Analysis , Y.H. Bauer, G.D. Christian, J.E. O'Reilly. Allyn and Bacon, Inc.
5. Introduction to Instrumental Analysis , R. D. Braun, PharmaMed Press.
6. Introduction to Spectroscopy, D. Pavia, G. Lampman, G. Kriz Thomson Brooks Cole
7. Modern Molecular Spectroscopy, H.S. Randhawa, Mcmillan India Ltd.
8. Electronic Absorption Spectroscopy and related Techniques; D.N. Sathyanarayana, Universities Press.
9. Chemical Kinetics, K.J. Laidler , Tata McGraw Hill Publishing Company 2nd Edition.
10. Principles of Instrumental Analysis, D.A. Skoog, F.J. Holler and T.A. Nieman 5th Edition (1998), Saunders College Publishing, Harcourt Brace

& Company, U.S.A.

11. Automation in the laboratory By William Jeffrey Hurst, VCH, 1995

M Sc Semester IV Analytical Chemistry

CHE2402AC Pharmaceutical Analytical Chemistry – 3 credits

Unit I a) *Pharmaceutical Analysis - I*

- Logistics in drug discovery, pharmaceutical legislation - Indian drugs and cosmetics act, ICMR guidelines, registration requirements for a new drug.
- objectives of clinical drug development, role of FDA in drug development, regulatory requirements for analytical method development of new drug, Introduction to pharmacopoeia and specifications on finished products, stability studies, expiry date fixation.

b) *Phytopharmaceuticals and Neutraceuticals*

- WHO guide lines of the standardization of Herbal raw materials and finished products.
- Extraction isolation and analysis of phytopharmaceuticals -A detailed study of various methods of extraction and isolation of phytopharmaceuticals -
- Determination of Physical and chemical constants.
- Preliminary Screening of Natural Products:
- Marine drugs

Unit II *Pharmaceutical Analysis-II*

- Sources of impurities in pharmaceuticals (chemicals, raw materials, methods of manufacture, reagents, solvents, atmospheric and microbial contaminants) and storage effects.
- Limit tests, solubility tests, disintegration tests, stability studies, impurity profile of drugs,
- Process analytical technology, quality audits etc. Validation of manufacturing and analytical equipments, process validation in production of pharmaceuticals, development of new analytical / bioanalytical method and its validation.

Unit III *Techniques of pharmaceutical analysis-I*

- Assay of main classes of pharmaceuticals-with reference to Introduction-Type-Properties-Method of Analysis
- Electrochemical Methods: Potentiometry and conductometry. Flame photometry, Atomic absorption spectrophotometry, nuclear magnetic resonance spectroscopy and mass spectrometry
- Spectroscopic Methods: Ultraviolet, visible, Infrared and Raman spectroscopy
- Chromatographic Methods: Ion exchange, column, paper, thin layer, gas and high performance liquid chromatography.

- Assay of polymorphs in pharmaceutical Industry- introduction to polymorphs, methods of identification and analysis of polymorphs using Near-IR, Raman, XRPD, solid state NMR, DSC, TGA
- Methods of analysis of common excipients /additives in formulations.
- Determination of active constituents in presence of these excipients.

.BOOKS RECOMMENDED

1. Pharmaceutical Analysis, T.Highchi and E. Brochmann, Hanssen Interscience.
2. The quantitative analysis of drugs, D.C. Garrent, Chapman and Hall.
3. Pharmaceutical Analysis, A.H. Backett and J.B. Stenlake, Chapman and Hall.
4. Methods of drug analysis, S .F .Granbowrshll, Lee and Fehiger .
5. Analysis of drug and chemicals, N. Evers, W. Smith and C. Grikin.
6. Isolation and identification of drugs~ E. G.J Clerks,Pharmaceutical Press.
7. Indian Pharmacopia
8. Pharmaceutical analysis, A textbook for pharmacy students and pharmaceutical chemists, David G.Watson, Churhill Livingstone, 2000
9. Quality control of Herbal Drugs, Dr. Pulok Mukherjee Business Horizon G59, Masjid Moth, Gk-2, New Delhi 2002
10. Herbal Drug Technology, Dr. S S Agrawal Orient Longman Pvt. Ltd.3-6-752 Himayat Nagar Hyderabad -29 2007
11. Modern Methods of Plant Analysis Peach K and Tracey M V Vol 1 - 4 , Narosa Publishing House, New Delhi
12. The Practical Evaluation of Phytopharmaceuticals, Brain K.R and Turner T.T Wright-Sciencetchnica, Bristil

M Sc Semester IV Analytical Chemistry

CHE2403AC Advanced Analytical Techniques-3 credits

Unit I *Bioanalytical Chemistry*

- Bioanalytical Chemistry: Strategies for logical sample preparation, removal of matrix components,
- Methods for characterization and quantitation of biomolecules
- Principles of Electrophoresis: Introduction, electrophoretic support media, effect of experimental conditions on separations, .
- Applications of zone electrophoresis, isoelectric focussing, capillary electrophoresis. Ultracentrifugation.
- Pharmacokinetics: Clinical pharmacokinetics, drug absorption, drug metabolism, Analysis of drugs/ metabolites in biological fluids

Unit II *Luminescence Spectroscopy and high frequency titrations*

- *Chemiluminescences* :Introduction, principles, types. Measurement of chemiluminescence, Quantitative chemiluminescence Gas phase chemiluminescence analysis.Chemiluminescences titrations.Electro chemiluminescence
- Instrumentation for Fluorescence Spectroscopy
- Atomic fluorescence spectroscopy, resonance ionization spectroscopy, laser enhanced ionization, principle, , detecting of various gases, liquid and solids, sources, cell, monochromators, detector.
- X-ray Fluorescence spectroscopy (XRF) : Introduction and basic theory; Instrumentation, spectral analysis; Analytical information and applications
- Applications of Fluorescence Phenomena
- High frequency titration Introduction: theory and instrumentation, high frequency titrimetry, types of cell.Advantage of high frequency methods, applications.

Unit III *Radioanalytical Methods of Analysis*

- Radiation detection and measurements. Ionization current measurements, multiplicative ion collector, methods not based on ion collection, Instrumentation Counting staticsits..
- Activation analysis: Neutron activation analysis, principle, technique, steps involved in neutron activation analysis. Radiochemical and instrumental methods of analysis, important applications of NAA.
- Isotope dilution analysis:Principle, types of isotope dilution analysis, typical applications of isotope dilution analysis.
- Radiometric titration: Principle, techniques based on complex formation and precipitation, radiometric titration curves for estimation of ions from their mixture.
- Radio pharmaceuticals

BOOKS RECOMMENDED

1. Bioanalytical Chemistry, By Susan R. Mikkelsen, Eduardo Cortón, Wiley Interscience, 2004
2. Analytical Biochemistry, D.J Holms and H.Peck, Longman, 3rd edition
3. Essentials of Nuclear Chemistry, H J Arnika, New Age Publishers (2005).
4. Fundamentals of Radiochemistry D.D.Sood A.V.R.Reddy and N.Ramamoorthy
5. Frontiers in Nuclear Chemistry D.D. Sood A.V.R. Reddy and P.K. Pujari
6. Instrumental Analysis, H H Bauer, G D Christian, J E O'Reilly, Allyn and Bacon Inc.(1977)
7. Introduction to Instrumental Analysis, R D Braun, McGraw-Hill International Editions.(1987)
8. Instrumental methods of analysis, H H Willard, L L Merritt, J N Dean and F A Settle, IBH Publishing House, New Delhi (1986)

M Sc Semester IV Analytical Chemistry

Practical Courses

Advanced Analytical Chemistry Practicals-3 CHE2404AC
Advanced Analytical Chemistry practicals-4 CHE2405AC

(5 credits)

Separation techniques

Separation of Iron and Nickel ions from mixture by solvent extraction and their determination

Determination of Nickel using DMG by molten-naphthalene extraction

Determination of Nickel using DMG by solvent extraction

Determination of copper using diethyl dithiocarbamate by solvent extraction

Determination of iron using oxine by solvent extraction

Determination of SiO₂, CuO and Fe in copper pyrite

Determination of Lead (Pb) and Tin (Sn) in solder

Separation and determination of Zinc and Magnesium on anion exchanger

Separation and determination of Zinc and Cadmium using ion exchanger

Determination of Cl⁻ and Br⁻ in a mixture by ion exchange method

Separation and determination of Cu and Fe in a given mixture

Separation and determination of Fe and U in a given mixture

Determination of ion exchange capacity of a given ion exchanger

Separation and determination of molecular weight of proteins by electrophoresis

Separation and determination of copper and cadmium by polarographically

Determination of phenol from soil sample by Soxhlet extraction

Determination of nitrogen content of given fertilizer sample by Kjeldahl method

Flue gas analysis by Orsat's apparatus

Water treatment

Determination of free chlorine and available chlorine in water

Determination of COD in a given water sample

Determination of phosphate in water spectrophotometrically in a given water sample.

Determination of ammonia in water spectrophotometrically

Determination of ammonia / phosphate in water by standard addition technique

Determination of dissolved oxygen in water

Determination of sulphate in water by turbidimetry

M Sc Semester 4
Analytical Chemistry

CHE2406AC
Comprehensive Viva Voce Examination (2 Credits)

Comprehensive Viva voce Examination by Internal and External Examiners at the end of the IV semester.

M Sc Semester 4
Analytical Chemistry

CHE2407AC
Dissertation (3 Credits)

Dissertation work. To be evaluated by a panel of internal examiners at the end of the IV semester.

M Sc Semester IV
Analytical Chemistry

M Sc Semester 4
Analytical Chemistry

Special Practical and Assignment
CHE2408AC(3 Credits)

M Sc Semester IV Analytical Chemistry

CHE2401AE Electroanalytical techniques – 2credits

Unit I Electroanalytical Methods I

- *Polarography* :Introduction, Interpretation of a polarographic curve. Instrumentation. Supporting electrolytes. Diffusion coefficient and its evaluation. Ilković equation and its applications. Criteria of polarographic reversibility. Half-wave potentials and their significance. Methods of quantitative analysis
- Sampled DC polarography (Tast), differential and derivative voltammetry, square wave polarography.

Unit II Electroanalytical Methods II

- Linear sweep voltammetry, cyclic voltammetry, Fast cyclic voltammetry, Potential step techniques, Chronoamperometry, Chronopotentiometry.
- Stripping analysis
- Hydrodynamic Voltammetry rotating disk and rotating ring electrodes oxygen electrode, glucose sensor, Amperometric titrations.
- Modified electrodes
- Spectro electrochemistry
- Impedance spectrometry

Books Recommended

1. Polarographic Techniques, L. Meites 2nd Edition (1965), John Wiley, NY.
2. Principles of Polarography, J. Heyrovsky and K. Kuta, Academic Press, New York.
3. Principles of Instrumental Analysis, D.A. Skoog, F.J. Holler and T.A. Nieman 5th Edition (1998), Saunders College Publishing, Harcourt Brace & Company, U.S.A.
4. Electrochemical Methods: Fundamentals and Applications, A.J. Bard and L.R. Faulkner 2nd Edition (2000), Wiley, New York.
5. Fundamentals of Electroanalytical Chemistry, P Monk, John Wiley, NY.

M Sc Semester IV Analytical Chemistry

CHE2402AE

Special topics in Environmental Chemistry-- 2 credits

Unit-I

Thermal Pollution.sources, effects and remedies to reduce thermal pollution

Noise pollution: Sources of noise pollution, measurement of noise and Indices, effect of meteorological parameters on noise propagation. Noise exposure levels and standards.

Radioactive Pollution: Types, sources and consequences. Sampling methods.

Biological effects of ionizing radiation's: ICRP recommendations;

Unit-II

Hazardous waste: Disposal of hazardous solid waste on land & its impact on soil pollution.Destruction of some hazardous substances

Hazardous Wastes Management and Handling Rules, 1989, Resource Management, Disaster Management and Risk analysis

Environmental problems at global level and in India

Books Recommended

1. Environmental Pollution analysis, S.M. Khopkar, New Age Publishers (2001).
2. Environmental Chemistry , S.K. Banerji, Prentice Hall of India Pvt. Ltd., New Delhi (1999).
3. Environmental Chemistry, Stanley Manahan, Lewis Publishers CRC Press, USA, (2005) 6th Edition.
4. Identification and Analysis of Organic Pollutants in water, L H Keith, Ann Arbor Science Publications (1976)

M Sc Semester IV Analytical Chemistry

CHE2403AE Nano materials – 2 credits

Unit I Synthesis of Nano materials

Introduction : Definition and terminologies, nano particle morphologies, size dependent properties like optical, electrical and magnetic properties.

Synthesis of nano materials

Physical methods: Mechanical methods, methods based on evaporation, laser ablation, sputtering, CVD, MBE.

Chemical methods: Quantum dots and 1D nanomaterials (chemical precipitation method, synthesis in confined media like reverse micelles, zeolites etc), synthesis from molecular precursors and molecular clusters, sol-gel method. 2D nanomaterials (L-B thin films, Self Assembled Monolayers, soft lithography).

3D nanomaterials and biological methods: Synthesis using microorganisms, use of natural templates like DNA. Biomimetic synthesis of nanoparticles.

Synthesis of graphemes and nanocomposites

Unit II *Techniques for Characterizing Nanomaterials and Applications of Nanomaterials*

Ellipsometry, and dual polarization interferometry (DPI) as methods used to obtain nano-gram masses, nano-scale thicknesses, and the optical properties of ultra-thin assemblies.

- Infrared spectroscopy of nanoassemblies. Attenuated-total reflection (ATR) and grazing incidence angle techniques. Reflection-absorption IR spectroscopy (RAIRS). Surface enhanced Raman spectroscopy (SERS).
- Imaging techniques: AFM, STM, SEM, EDX, imaging ellipsometry..
- Optical microscopy. Fluorescence/phase contrast microscopy, confocal microscopy, total internal reflection microscopy (TIRM), Brewster angle microscopy (BAM),
- Nano-optics and local spectroscopy. Scanning near-field optical microscopy and photon scanning tunneling microscopy, scanning plasmon near-field optical spectroscopy (SPNM), near-field optical spectroscopy, fluorescence spectroscopy, nearfield nonlinear optics.
- Size determination: DLS, XRD, TEM

Applications of nanomaterials: Optical and bio sensors, Energy cell, Catalysis, Drug delivery systems, Waste remediation

Risk assessment and management of nano particles

Books Recommended

1. Chemistry of nanomaterials, Volume I & II, Ed. C N R Rao, A. Muller, A K Cheetham Wiley, VCH.(2004).
2. Introduction to Nanotechnology C P Poole Jr., F J Owens, Wiley (2006)
3. Nanotechnology, M. Ratner and D. Ratner. Pearson.

M Sc Semester IV Analytical Chemistry

CHE2404AE

Analysis of Industrial Materials - 2 credits

Unit I Fuel and Petrochemical Analysis

Fuels: Definition, classification and characteristics of fuels. Sampling.

Solid fuels; Proximate and ultimate analysis of coal, determination of calorific value.

Liquid fuels; Analysis of crude oil, Fractionation of crude oil and analysis of various fractions by GC. Cracking of naphtha and estimation of various olefins.

- Analysis of the final products (moisture, distillation range, density, flash point, fire point, smoke point, cloud point, aniline point, pour point, kinematic viscosity, Cu corrosion, Reid vapor pressure, carbon residue), component analysis by GC, composition analysis (PIONA). Octane and cetane numbers. Analysis of Biodiesel
- Gaseous fuels. Analysis of natural gas, liquefied petroleum gas, coal gas, water gas, producer gas, gober gas and blast furnace gas. Calorific value determination.
- *Petrochemicals:* Analysis of naphtha and other feed stocks. Characterization of the catalyst used for cracking.

Unit II Analysis Industrial Materials

- *Analysis of Fertilizers:* Sampling and sample preparation, analysis of nitrogen, phosphorus and potassium.
- *Analysis of ceramics and glass :* Sodium and potassium by flame photometry, analysis of lead and borate glasses,.
- *Analysis of cosmetics:* General formulations, Analysis of deodorants and antiperspirants, Al, Zn, boric acid, chloride, sulphate, urea. Analysis of face powder: fats, fatty acid, Ca, Mg, BaSO₄, Ti and Fe. Oxides of Ti, Fe and Al (total). Analysis of hair tonic preparations: 2, 5-diaminotoluene, KBrO₃, resorcinol, salicylic acid. Analysis of vanishing cream
- *Analysis of alloys:* Analysis of Cr, Mn, Fe, Co, Ni, and Zn in alloys
- *Analysis of Portland cement*
- *Analysis of paints and pigments:* Preliminary inspection of sample, Tests on the total coating, Separation of Pigment, Binder and Thinner of Latex Paints, Separation of Pigment, Binder and Thinner of Solvent Type Coatings, Identification of Binder, Identification and Analysis of Pigments, Identification and Analysis of Thinner
- *Analysis of pesticide Residues* Classification of pesticides, environmental degradation of pesticides, sample preparation and methods of analysis of pesticide residues

BOOKS RECOMMENDED

1. Standard methods of chemical analysis , F.J. Welcher, Van Norstrand, 1927
2. Applied Chemistry. Theory and Practice ,O.P. Virmani and A.K. Narula. 2nd Edition.
3. Metallurgical Analysis, B.C. Agrawal and S.P. Jaub, Khanna Publishers.
4. Standard methods of Chemical Analysis Vol. I and II, W. W. Scott. D. Van Nostrand Co. Inc. Princeton N. York.
5. Encyclopedia of Industrial Chemical Analysis Vol I-20, F.D. Shell and D.S. Effie.
6. Colour and constitution. John Griffiths , Wiley and Sons, 3rd Edition.
7. Official Methods of Analysis of the Association of official Analytical Chemists (AOAC), 14TH Ed. (1984).
8. Insight into Speciality Inorganic Chemicals, Ed. David Thomson (1995) RSC
9. Handbook of pesticides: methods of pesticide residues analysis, [Leo M. L. Nollet](#), [Hamir Singh Rathore](#), CRC Press/Taylor & Francis, 2009
10. Vogel A, "Textbook of Quantitative Inorganic Analysis", Pearson Education Ltd