

ANDHRA UNIVERSITY
DEPARTMENT OF CHEMISTRY
M.SC., CHEMISTRY (FINAL)-III-SEMESTER SYLLABUS
SPECIALIZATION: ANALYTICAL CHEMISTRY
PAPER - I: SEPARATION METHODS - I
(With effect from the admitted batch of 2021-2022 academic year)

Unit - I Chromatography - 1

Chromatography: classification of different chromatographic methods, methods of development-Elution development, Gradient elution development, displacement development, and frontal analysis.

Principles of chromatography, different migration, adsorption phenomena, partition, adsorption coefficient, retardation factor, retention time and volume, column capacity, temperature effects, partition isotherm.

Unit - II Chromatography - 2

a. Dynamics of chromatography-efficiency of chromatographic column, zone spreading, High Equivalent Theoretical Plate (HETP), Van Demeter equation, resolution, choice of column, length and flow velocity, qualitative and quantitative analysis.

b. **Paper chromatography:** Principle, papers as a chromatographic medium, modified papers, solvent systems, mechanism of paper chromatography, experimental technique, different development methods-ascending, descending, horizontal, circular spreading, multiple development, two dimensional development, reverse phase paper chromatographic technique-visualization and evaluation of chromatograms, applications.

Unit - III Chromatography - 3

a. **Thin layer chromatography:** principle, chromatographic media-coating materials, applications, activation of adsorbent, sample development, solvent systems, development of chromatoplate, types of development, visualization methods, documentation, and applications in the separation

b. **High Performance Thin layer chromatography (HPTLC):** principle, technique, and applications.

c. **Column chromatography (adsorption chromatography):** principles, general aspects, adsorption isotherms, chromatographic media, nature of forces between adsorbent and solutes, eluents (mobile phase), column chromatography without detectors and liquid chromatography with detectors and applications.

Unit – IV Chromatography – 4

- a. Gel Exclusion chromatography or Gel filtration chromatography:** principles, properties of xerogels, apparatus and detectors, resolution of gel type, applications to organic compounds.
- b. Capillary Electrophoresis:** Principle, Details of the Instrument, Applications to Inorganic and Organic compounds.
- c. Inorganic molecular sieves:** structure of zeolites, crystals, types of sieves, application in the separation of gases including hydrocarbons, ion exclusion-principles and applications,

Unit – V Chromatography – 5

- a. Gas chromatography:** Theory, Instrument description of equipment and different parts, columns (packed and capillary columns), detector specifications-thermal conductivity detector, flame ionization detector, electron capture detector, nitrogen-phosphorus detector, photo ionization detector, programmed temperature gas chromatography; applications in the analysis of gases, petroleum products etc., other detectors used their Principles and Applications.
- b. GC-MS – Introduction**
Instrumentation – GC – MS interface – Mass spectrometer (MS) Instrument Operation, processing GC – MS data – ion chromatogram Library searching – Quantitative measurement – sample preparation Selected ion monitoring – Application of GC-MS for Trace constituents. Drugs analysis, Environmental Analysis and others.
- c. Counter current chromatography-principles and application, Affinity chromatography- principles and applications**

Text books:

1. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
2. M.N. Sastri, Separation methods, Himalaya Publishing Company, Mumbai

Reference books:

1. E. Helfman, Chromatography, Van Nostrand, Reinhold, New York
2. E. Lederer and M. Lederer, Chromatography, Elsevier, Amsterdam.
3. Chemical separation methods, John A Dean, Von Nostrand Reinhold, New York
4. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
5. H.M Mc Nair and J. M. Miller, Basic Gas Chromatography, John Wiley, New York
6. W. Jeumings, Analytical Gas chromatography, Academic Press, New York
7. H. Eugelhardt (ed), Practice of HPLC, Springer Verrag, Berrin.

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DEPARTMENT OF CHEMISTRY
M.SC., CHEMISTRY (FINAL)-III-SEMESTER SYLLABUS
SPECIALIZATION: ANALYTICAL CHEMISTRY
Paper- II: Quality control and Traditional methods of Analysis-I
(Effective from admitted batch 2021-2022)

Unit – I: Quality control in Analytical Chemistry

- a. **Characteristics of an analysis:** quality of an analytical procedure, limit of detection, sensitivity, safety, cost measurability, selectivity and specificity, quality control-principles of Ruggedness test, control charts, Youden plot, and ranking test.
- b. **Quality assurance and management systems:** elements of quality assurance, quality assurance in design, development, production and services, quality and quantity management system, **ISO 9000** and **ISO 14000** series-meaning of quality, quality process model, customer requirement of quality calibration and testing, statistical process control, process control tools, control chart, statistical quality control, acceptance sampling.
- c. **Evaluation and reliability of analytical data:** limitation of analytical methods, accuracy, precision, errors in chemical analysis, classification of errors, minimization of errors, significant figures, computations and propagation of errors.
- d. Brief out line of ICH guide lines on drug substances and products.

Unit – II

- a. **Statistical analysis:** Mean deviation, Standard deviation, coefficient of variance, normal distribution, F test, T test, rejection of results, presentation of data.
- b. Good laboratory practices (GLP) – need for GLP, GLP implementation and organization, GLP status in India.
- c. **Decomposition and Dissolution of Inorganic Compounds-**Principle of decomposition and Dissolution. Difference between dissolution/decom-position of Organic and Inorganic substances. Importance of Decomposition Techniques in Analysis. Principle of Dissolution of an inorganic substance. Decomposition of samples with acids – H₂O, HCl, HF, HNO₃, H₂SO₄ and HClO₄

Unit – III

- a. Decomposition of samples by fusion, Principle and with two examples each

Alkali Fusion--- Na_2CO_3 , NaOH ,

Acidic Fusion--- Sodium Hydro Sulphate, Sodium Pyro Sulphate

Oxidation Fusion--- Na_2O_2 , Sodium Chlorate

Reductive Fusion $\text{Na}_2\text{CO}_3 + \text{Na}_4\text{BO}_4$

What is Sintering process, How is it different from Fusion.

Fusion with alkali carbonates, alkali hydroxides, Sodium Peroxide

Decomposition of samples by sintering with sodium peroxide, sodium carbonate.

Principles of decomposition at high temperatures, high pressures .

Principles of Microwave and ultrasonic decomposition techniques.

b. Organic Compounds

Principles of solubility of organic compounds, non polar, polar solvents.

Recrystallisation methods and application of solubility and Recrystallisation.

Unit – IV Oxidant systems – Principles and applications in analysis

Analytical chemistry of some selected oxidant systems – formal, standard and normal potentials in various media, species responsible for the oxidation properties, stability of the solutions, standardization, requirement for the selections of the oxidants, selection of suitable indicators for Oxidant systems.

a) Inorganic Systems Mn (III), Mn (VII), Ce (IV), Cr (VI), V (V), periodate, iodate,

b) Organic Systems chloramine-T.

Unit – V Organic Functional group analysis

Classification of functional groups with suitable examples.

Determination of:

- 1) Functional groups imparting acidic nature – thiol, enediol, phenolic hydroxyl.
- 2) Functional groups imparting basic nature – Aliphatic and Aromatic primary, secondary and tertiary amines – hydrazine derivatives.
- 3) Functional groups which impart neither acidic nor basic nature – Aldehydes, Ketones, Nitro, Methoxy, Olifinic.

Text books:

1. Technical methods of analysis – Griffin, McGraw Hill Book Co.
2. Chemical Separation and measurements – D.G Peterseti, John M.Haves Sanders Co.
3. Chemical analysis – H.A Laitinan, McGraw Hill Book Co.
4. Newer redox titrants – Berka, Zyka and Vulterin, Pergamon Press
5. Volumetric Analysis, Vol III – I.M Kolthoff and R. Belcher, Interscience Public, New York

6. Vogel's Text Book of Inorganic Quantitative Analysis – J. Bassett et al, ELBS
7. Organic functional groups – S. Siggia.

Reference Books:

1. D.A Skoog, D.M West and F.J Holler, Analytical Chemistry, An Introduction, Sanders College Publishing, New York
2. K.V.S.G Murali Krishna, An Introduction ISO 9000, ISO 1400 Series, Environmental Management
3. Quality Assurance and Good Laboratory Practices, Prof. Y. Anjaneyulu, In Now Publication, New York
4. Quality Assurance in Analytical Chemistry – G.Kateman and F.W Pijpers, John Wiley and Sons, New York
5. Quantitative Chemical Analysis – I.M Kolthoff, E.B Sandel, E.J Meehan, S. Bruckenstein, Macmillan Company, London
6. Decomposition Techniques in Inorganic Analysis – J.Dolezal, P.Povondra, Z.Sulcek

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SPECIALIZATION: ANALYTICAL CHEMISTRY
Paper – III: Applied Analysis-I
(Effective from 2021 - 2022 Admitted batch)

Unit – I Analysis of Ores - I

- (a) General techniques of analysis applied to complex materials - Scope of metallurgical analysis -
General methods of dissolution of complex materials - Various chemical methods for the effective separation of the constituents in the complex materials.
- (b) Analysis of ores: Iron ore- Analysis of the Constituents – Moisture , loss of ignition, Total Iron, ferrous Iron ,Ferric Iron, alumina , silica, Titania, Lime, Magnesia, Sulphur, phosphorous, manganese, alkalies, combined water, Carbon in blast furnace, flue dust and sinter.
- (c) Manganese Ore - Analysis of the Constituents – Total Manganese, MnO_2 , SiO_2 , BaO , Fe_2O_3 , Al_2O_3 , CaO , P and S

Unit – II
Analysis of Ores - II

- (a) Chromite Ore - Analysis of the Constituents – Chromium, SiO_2 , FeO , Al_2O_3 , CaO , & MgO .
- (b) Phosphate rock Ore - Analysis of the Constituents - CaO , P_2O_5 , F, SiO_2 , CO_2 , S, Na_2O , Al_2O_3 , Fe_2O_3 , MgO , K_2O , Cl , MnO . Organic carbon, Moisture, Loss of ignition.
- (c) Aluminium Ore (Bauxite) - Analysis of the Constituents – Silica, Alumina, Fe_2O_3 , Titania, MnO , P_2O_5 , CaO , MgO , vanadium, zirconium, and alkalies.

Unit – III
Analysis of Finished Products – I

- (a) Analysis of steel for C, Si ,S, P, Mn, Ni, Cr; Mg and analysis of blast furnace slag .
- (b) Analysis of refractory materials: fire clay, flour spar, and magnesite
- (c) Analysis of fluxes - limestone and dolomite.

Unit – IV

Analysis of Finished Products – II

- (a) Chemical Analysis of cement-silica, NH_4OH group, ferric oxide, alumina, lime, magnesia, SulphideSulphur, K_2O , Na_2O , free CaO in Cement and Clinker, SO_3 and loss on ignition.
- (b) Analysis of oils - saponification number, iodine number, and acid number..
- (c) Analysis of soaps - moisture, volatile matter, total alkali, total fatty matter, free caustic alkali or free fatty acids, sodium silicate, chloride.
- (d) Analysis of paints-vehicle and pigment, BaSO_4 , total lead and lead chromate

Unit – V

Assessment of water Quality

Sources of water, classification of water for different uses, types of water pollutants and their effects,

Analytical methods for the determination of the following ions in water:

Anions: CO_3^{2-} , HCO_3^- , F^- , Cl^- , SO_4^{2-} , PO_4^{3-} , NO_3^- , NO_2^- , CN^- , S^{2-}

Cations: Fe^{2+} , Fe^{3+} , Ca^{2+} , Mg^{2+} , Cr^{3+} , As^{5+} , Pb^{2+} , Hg^{2+} , Cu^{2+} , Zn^{2+} , Cd^{2+} , Co^{2+}

Determination of Dissolved oxygen (D.O), Biochemical Oxygen Demand (BOD) and

Chemical Oxygen Demand (COD), standards for drinking water.

Text books

1. Handbook of Analytical Control of Iron and Steel Production, Harrison John, Wiley 1979
2. Standard methods of Chemical Analysis, Welcher
3. Technical Methods of Analysis, Griffin, McGraw Hill
4. Commercial Methods of Analysis, Foster Dee Sneel and Frank M. Griffin, McGraw Hill Book Co.
5. Water Pollution, Lalude, McGraw Hill
6. Environmental Chemistry, Anil Kumar De, Wiley Eastern Ltd.
7. Environmental Analysis, S.M. Khopkar (IIT Bombay)

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SPECIALIZATION: ANALYTICAL CHEMISTRY
Paper – IV: INSTRUMENTAL METHODS OF ANALYSIS – I
(Effective from 2021-2022 Admitted batch)

Unit – I :

(a) UV-Visible Spectroscopy: Introduction to UV-Visible spectroscopy, deviation from

Beer's law; Instrumentation- single and double beam spectrophotometers, sources of radiation, filters and monochromators, sample cells, detectors. Principle of diode array spectrophotometers, Principle of UV-visible diffuse reflectance spectrophotometer, spectrophotometric titrations, applications- general precautions in colorimetric determinations, colorimetric determination of Fe^{2+} , Fe^{3+} , Al^{3+} , NH_4^+ , Cr^{6+} , Co^{3+} , Cu^{2+} , Ni^{2+} , NO_2^- and PO_4^{3-} using suitable reagents, simultaneous determinations of dichromate and permanganate in a mixture, determinations of aromatic primary amines, riboflavin and paracetamol.

Unit – II:

(a) Spectrofluorimetry: Theory of fluorescence, and phosphorescence, types of fluorescence, factors affecting fluorescence intensity, relationship between fluorescence intensity and concentration; Quenching of fluorescence and types of Quenching, Instrumentation, advantages and limitations of fluorimetry. Applications-determination of Al^{3+} , chromium salts, Li^+ , fluorescence, thiamin (B1) and riboflavin (B12), nicotinamide and methyl dopa.

(b) Raman Spectroscopy: Theory, instrumentation, sample handling, instrumentation, Raman spectra of HCN , CO , CO_2 , N_2O , H_2O and quantitative analysis.

Unit – III:

Infrared Spectroscopy: Introduction to Infrared Spectroscopy, characteristic frequencies of organic compounds, instrumentation – IR sources, **sample cells**, sample preparations techniques, detectors; dispersive IR spectrophotometer, Non-dispersive IR spectrophotometer for analysis of *CO and other* organic compounds, principle of FTIR spectrometer, Attenuated Total Reflection (ATR) IR. Qualitative and quantitative analysis with reference to petroleum industry and polymer industry, Identification of drug substances and identification of impurities in a drug sample.

Unit – IV:

(a) NMR Spectroscopy: Introduction to NMR spectroscopy-resonance condition, nuclei with quadruple moments, relaxation process and causes for relaxation process (Magnetic dipole-dipole interactions, paramagnetic relaxation, chemical shift anisotropy, quadruple relaxation, scalar relaxation and spin rotation relaxation), chemical shift, factors affecting chemical shift, shielding, spin-spin splitting, mechanism for spin-spin coupling, factors affecting the germinal and vicinal coupling, NMR Instrumentation, sample preparations, FT NMR, Solid state magic angle NMR; **spin-decoupling experiments**-double resonance, off-resonance, spin tickling, nuclear over house effect and shift reagents; NMR of other than proton- ^{13}C , ^{15}N , ^{19}F , ^{31}P , and ^{11}B . Variable temperature NMR experiment for fast chemical reactions and fluxional molecules.

(b) ESR Spectroscopy: principle, g value, hyper fine splitting, qualitative analysis, Kramer's degeneracy, fine splitting, instrumentation, introduction to double resonance technique, difference between ESR and NMR spectra, ESR spectra of free radicals and other analytical applications.

Unit – V:

(a) Mass Spectroscopy: Principle, types of peaks observed, resolution, **Instrumentation-sample inlet systems** (batch inlet, direct probe, chromatographic and capillary electrophoretic), **Ionization techniques:** Electron impact ionization, Electrospray ionization, Chemical ionization, atmospheric pressure chemical ionization, Matrix-assisted laser desorption ionization, fast atom bombardment and inductive coupled plasma; **Mass Analyzers:** Magnetic sector mass analyzer (single and double focusing), Quadruple, Ion traps, Time of flight (TOF); **Ion detectors**, FTMS, Tandem mass spectrometry(MS^n), qualitative analysis, molecular weight determination, quantitative analysis.

(b) X-Ray Spectroscopy (XRF): basic principle, Instrumentation- energy dispersive and wavelength dispersive techniques, chemical analysis by X-ray spectrometers, matrix effects, applications.

Text Books:

1. Instrumental methods of analysis – H.H Willard, Meritt Jr. and J.A Dean
2. Principles of instrumental analysis – Skoog and West
3. Vogels Textbook of Quantitative Inorganic analysis – J. Basset, R.C Denney, G.H Jefferey and J.Madhan
4. Instrumental methods of analysis – B.K Sarma, Goel Publishing House, Meerut
5. Instrumental methods of Analysis – Ewing

6. Handbook of ICP- Bogdain B.
7. Analytical Chemistry Instrumental techniques, Maninder Singh, Dominant Publishers, New Delhi, 2002.
8. Mass spectrometry for Chemists and Biochemists, Robert A.W. Johnstone and Macolm.E. Rose, 2th ed Cambridge University Press 1996.
9. Structural methods in Inorganic chemistry - E.A.V. Ebsworth, et al ELBSPublications, 1988
10. Physical methods for Chemists, Russell S. Drago second edition, Saunders College publishing 1992.
11. Mass Spectrometry Basics, Herbert, Christopher G.; Johnstone, Robert A.W., CRC Press.

Reference Books:

1. Applications of ICP-MS, A.R Date and A.L Glay, London (Eds), Blackie, London
2. A. Moutaser and D.W Gologhtly (Eds), ICP in Analytical Atomic Spectrometry, VeH Publishers, New York
3. G.I Moore, Introduction to ICP emission Spectrometry in Analytical Spectroscopy, Elsevier, Amsterdam
4. Infrared and Raman Spectra of Inorganic and Coordination Compounds, Kazuo Nakamoto, 5th ed., John Wiley & Sons, 1995.
5. Instrumental methods of Analysis – Chatwal and Anand
6. 16. Mass Spectrometry-A Textbook by Jürgen H. Gross, © Springer-Verlag Berlin Heidelberg 2004, Printed in Germany.

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SPECIALIZATION: ANALYTICAL CHEMISTRY
ANALYTICAL CHEMISTRY PRACTICAL - I
(With effect from 2021-2022 admitted batch)

CLASSICAL METHODS OF ANALYSIS-1

1. Water analysis
 - (i) Determination of total hardness (Ca^{2+} and Mg^{2+}) of water samples
 - (ii) Determination of chloride (Cl^-) present in water samples
 - (iii) Determination of dissolved oxygen (DO) of drinking water and sewage water

2. Complex metric titrations
 - (i) Determination of the concentration of calcium in milk powder by complexometric titration (EDTA)
 - (ii) Determination of Calcium and Magnesium in limestone or dolomite samples using EDTA.

3. Fertilizer analysis
 - (i) Determination of ammonia from ammonia containing fertilizer
 - (ii) Determination of phosphate from fertilizer

4. Analysis of iron ore
 - (i) Complete analysis of iron ore
 - (ii) Determination of percentages of Fe (II) and Fe (III) present in iron ore sample

5. Analysis of Coal
 - (i) Determination of moisture content of coal sample
 - (ii) Determination of volatile matter of coal sample
 - (iii) Determination of fixed carbon of coal sample
 - (iv) Determination of ash content of coal sample

INSTRUMENTAL METHODS OF ANALYSIS-1

1. pH metry
 - (i) Determination of alkalinity of a coloured effluent using pH metric titration.
 - (ii) Determination of purity of commercial HCl using pH metric titration.
 - (iii) Determination of purity of commercial H_2SO_4 using pH metric titration.
 - (iv)

2. Conductometry

- (i) Determination of concentration of strong acid using strong base
- (ii) Determination of concentration of weak acid using strong base
- (iii) Determination of sodium carbonate using strong base

3. Potentiometry

- (i) Determination of Cr(VI) with Fe(II) using potentiometric end point
- (ii) Determination of Fe (II) using ceric sulphate by potentiometric end point
- (iii) Determination of a mixture of Ce(IV) and V(V) with Fe(II) by potentiometric end point
- (iv) Determination of KSCN with AgNO₃ by potentiometric end point.

4. Spectrophotometry

- (i) Determination of Fe (III) using potassium thiocyanate
- (ii) Determination of Iron(II) using orthophenanthroline
- (iii) Determination of phosphate in fertilizer and cola drinks by Molybdenum blue method
- (iv) Determination of Manganese (II) -periodate method

5. Flame photometry

- (i) Determination of sodium present in bread samples
- (ii) Determination of sodium and potassium in a given sample of fertilizer

6. Thin layer chromatography: Determination of R_f values and identification of organic compounds in a given mixture by TLC

- (i) Separation of mixture of benzil and 2-nitrophenol
- (ii) Mixture of benzophenone and naphthalene
- (iii) Mixture of 2-nitrophenol and 4-nitrophenol

References:

1. A Text Book of Quantitative Inorganic Analysis (3rd Edition) – A. I. Vogel

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SPECIALIZATION: ANALYTICAL CHEMISTRY
PAPER - I: SEPARATION METHODS – II
(With effective from 2021-2022 admitted batch)

Unit – I

- (a) **Ion Exchange:** principles of ion-exchange systems, synthetic ion-exchange resins, properties of anion and cation exchange resins, ion-exchange mechanism, ion-exchange equilibria, selectivity, ion-exchange capacity, applications of ion-exchangers in different fields.
- (b) **Ion exchange chromatography:** Principle, Equipment, Application Specifically Separations of Lanthanides, Actinides, amino acids.
- (c) **Ion chromatography:** principles of separation, instrumentation, detectors, separation of cations and anions, applications in the analysis of water and air pollutants.

Unit – II

High performance liquid chromatography: Theory, Instrument description of the different parts of the equipment, columns, detectors-UV detector, refractometric detector, Fluorescence detector, Diode Array detector, applications in the separation of organic compounds, names of other detectors used their Principles and Applications.

LC-MS– Introduction – Instrumentation – liquid chromatograph – Mass spectrometer Interface – Instrumental details – Processing LC-MS data – ion chromatograms – Library searching – Quantitative measurements. Sample preparation – selected ion monitoring. Application of LC-MS for Drug analysis, Environmental samples and others.

Unit - III

- d. **Liquid-liquid partition chromatography:** principle supports, partitioning liquids, eluents, reverse phase chromatography, apparatus and applications
- e. Development and validation of an analytical method, units, concentrations, calculations, standards, chemical reactions, expressions of concentrations, importance of separation methods with examples.
- f. **Sampling:** Basis of sampling, purpose of sampling, homogeneous and heterogeneous samples, statistical criteria for good sampling, sample size, sampling unit, gross sample, laboratory sample.

Unit – IV

Sampling of Solids: Cone and Quartering method, Long pile and alternative shovel method, precautions in preservation of solid samples, sampling of metals and other solids rods, wires, sheets, plates, especially Gold, Silver, Iron and other metals. Sampling of different types of liquids: different sampling techniques, sampling of drinking water, industrial effluents, precautions in sampling and preservation of collected liquid samples. **Sampling of gases:** sampling and Pre-concentration by adsorption or absorption method, instantaneous monitoring, sampling in samplers and subsequent monitoring, different types of gas samplers, precautions in preservation of samples, systematic sampling and random sampling.

Unit – V

- (a) **Importance of Analytical Chemistry to Industrial Research:** Importance of Qualitative and Quantitative analysis in research and development, industries and other branches of science.
- (b) **Solvent Extraction:** principles and processes of solvent extraction, Distribution Law and Partition coefficient, nature of partition forces, different types of solvent extraction systems – Batch extraction, Continuous extraction, Counter current extraction, solvent extraction systems, applications in metallurgy, general applications in analysis and pre-concentration, special extraction systems like crown ethers, super fluid and surfactant extractions-examples.

Text books:

1. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
2. M.N. Sastri, Separation methods, Himalaya Publishing Company, Mumbai

Reference books:

1. E. Helfman, Chromatography, Van Nostrand, Reinhold, New York
2. E. Lederer and M. Lederer, Chromatography, Elsevier, Amsterdam.
3. Chemical separation methods, John A Dean, Von Nostrand Reinhold, New York
4. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
5. H.M Mc Nair and J. M. Miller, Basic Gas Chromatography, John Wiley, New York
6. W. Jeumings, Analytical Gas chromatography, Academic Press, New York
7. H. Eugelhardt (ed), Practice of HPLC, Springer Verrag, Berrin

M.Sc. DEGREE EXAMINATION (Model Question Paper)

M.Sc.(Final) Chemistry-IV semester

Specialization – ANALYTICAL CHEMISTRY

Paper I –SEPARATION METHODS -II

Time: Three Hours

Maximum: 80 marks

SECTION - A

Answer ALL Questions

(5 x 16 = 80 marks)

1. (a) i) Give the principles of separation in ion chromatography.
ii) Explain the principle and equipment in ion exchange chromatography. Discuss its application in the separation of amino acids.
Or
(b) Explain the following: i) anion and cation exchange resins, ii) ion-exchange Mechanism, iii) ion exchange capacity and iv) regeneration of ion exchange resins.
2. (a) i) Discuss in detail the principle, instrumentation, advantages and applications of HPLC.
ii) Why liquid chromatography is a good technique for the separation of protein and nucleosides?
Or
(b) i) Explain the instrumentation of LC-MS
ii) Discuss the applications of LC-MS in analysis of drug and environmental samples.
3. (a) i) Explain the different types of concentration units
ii) Write the parameters for analytical method development and validation
Or
(b) i) Discuss the basis and purpose of sampling.
ii) Differentiate the stratified and random sampling.
4. (a)i) Discuss in detail about sampling of different types of liquids and preservation of Collected liquid samples
ii) Explain different methods for instantaneous monitoring of gases.
Or
(b) i) Explain different methods for sampling of solids.
ii) Write different types of gas samplers
5. (a)i) Give the importance of qualitative analysis in R&D and industries.
ii) What are crown ethers and explain their use in solvent extraction?
Or
(b)i) Give the principles and processes of solvent extraction and show that multiple extraction is better than a single extraction.
ii) Write short notes on batch extraction, continuous extraction and counter current extraction.

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Unit – I: Precipitation methods-I

- (a) Crystal habit and super saturation, nucleation and crystal growth, homogeneous and heterogeneous nucleation, solubility and particle size, colloids, completeness of precipitation, effect of excess precipitant, pH, complex formation, temperature, purity of precipitates, aging.
- (b) **Co-precipitation and post precipitation** : theory of adsorption of salts having an ion in common with the main precipitate, co-precipitation in colloidal precipitates, adsorption of solvents, mixed crystal formation by occlusion and entrapment, re-precipitation with examples, Post-precipitation – theory of post-precipitation, examples of post-precipitation, conditions for obtaining pure and quantitative precipitates.

Unit –II: Precipitation methods-II

- a. **Precipitation Titrations**: Principle, Indicators for precipitation titrations, determination of halides.
- b. **Precipitation from Homogeneous Solution (PFHS)**: theory of PFHS, methods of PFHS – increase in pH, decrease in pH, cation release, anion release, reagent synthesis, change in oxidation state, photochemical reactions, precipitation from mixed solvents. Applications of PFHS methods.

Unit –III: Gravimetric determinations

- a. **Gravimetric determinations**: nature of species, preparation of solutions, limitations, interferences, inorganic precipitants-chloride and sulphate, organic precipitants dimethyl glyoxime (DMG), oxine, benzidine, salicylaldehyde, benzoin oxime, sodium tetraphenylboron, tetraphenylarsonium chloride.
- b. **Electro-gravimetric analysis**: principle, important terms in electrogravimetry, decomposition voltage or decomposition potential, over voltage and their importance, instrumentation, electrolysis at constant current, determination of Cu^{2+} by constant current electrolysis, electrolysis at controlled potentials, determination of Cu, Pb, Sn in brass and bronze by controlled potential electrolysis.

Unit –IV

Analytical chemistry of some selected reductant systems – formal, standard and normal potentials in various media, stability of the solutions, species responsible for the reduction properties,

standardization, requirement for the selection of the reductants, selection of suitable indicators for various reductant systems,

(a) Inorganic Systems – Cr (II), V (II), Ti (III), Sn (II), Fe (II) in H_3PO_4 and hydrazine,

(b) Organic Systems – hydroquinone and Ascorbic acid.

Unit –V

Basic considerations of drugs – Classification

Determination of the following Drugs:

- 1) Acetyl salicylic acid (Antipyretic – Analgesic)
- 2) Testosterone, progesterone and cortisone (Steroids and corticoids)
- 3) Sulphadiazine(sulphadiazine)
- 4) Phenobarbitone (Barbituric acid derivatives)
- 5) Chloramphenicol, Benzyl penicillin and Tetracycline (Antibiotics)
- 6) Thiamine (B1), Riboflavin (B2) and ascorbic acid (c) [Vitamins]
- 7) Isoniazid (Antimicrobial agents)
- 8) Methyldopa (Antihypertensive agents)
- 9) Metronidazole (Antiamoebic agents).

Text books:

8. Technical methods of analysis – Griffin, McGraw Hill Book Co.
9. Chemical Separation and measurements – D.G Peterseti, John M.Haves Sanders Co.
10. Chemical analysis – H.A Laitinan, McGraw Hill Book Co.
11. Newer redox titrants – Berka, Zyka and Vulterin, Pergamon Press
12. Volumetric Analysis, Vol III – I.M Kolthoff and R. Belcher, Interscience Public, New York
13. Vogel's Text Book of Inorganic Quantitative Analysis – J. Bassett et al, ELBS
7. Organic functional groups – S. Siggia

Reference Books:

7. D.A Skoog, D.M West and F.J Holler, Analytical Chemistry, An Introduction, Sanders College Publishing, New York
8. K.V.S.G Murali Krishna, An Introduction ISO 9000, ISO 1400 Series, Environmental Management
9. Quality Assurance and Good Laboratory Practices, Prof. Y. Anjaneyulu, In Now Publication, New York
10. Quality Assurance in Analytical Chemistry – G.Kateman and F.W Pijpers, John Wiley and Sons, New York
11. Quantitative Chemical Analysis – I.M Kolthoff, E.B Sandel, E.J Meehan, S. Bruckenstein, Macmillan Company, London
12. Decomposition Techniques in Inorganic Analysis – J.Dolezal, P.Povondra, Z.Sulcek

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SPECIALIZATION: ANALYTICAL CHEMISTRY
Paper – III: Applied Analysis – II
(Effective from 2021-2022 Admitted batch)

Unit – I Analysis of raw materials - I

Analysis of non-ferrous alloys:

- (i) Brass – Analysis of the constituents – Cu, Zn, Sn, Pb and Fe.
- (ii) Bronze - Analysis of the constituents – Cu, Sn, Zn, Pb and Fe.
- (iii) Solder - Analysis of the constituents – Sn, Pb and Sb.

Unit – I Analysis of raw materials – II

Analysis of Ferro alloys :

- (i) Ferro silicon - Analysis of the constituents – Si, C, P, S
- (ii) Ferro vanadium - Analysis of the constituents – V, C, P, S, Si, Al.
- (iii) Ferro manganese - Analysis of the constituents – Mn, S, C, P, Si
- (iv) Silico manganese - Analysis of the constituents – Mn, S, C, P, Si
- (v) Ferro chromium - Analysis of the constituents – Cr, C, Si.

Unit – III Analysis of Soil, Fertilizer and Fuel

(a) Analysis of soils: sampling, determination of moisture, total N, P, Si, lime, humus nitrogen, alkali salts, soil absorption ratio.

(b) Analysis of fertilizers: ammonical fertilizers, Phosphate fertilizers, Nitrate fertilizers.

(c) Analysis of fuels: solid fuels-coal, proximate analysis, ultimate analysis, heating value, grading of coal based on Ultimate Heat Value (UHV).

Unit – IV Assessment of Air Quality

Composition of pure air, classification of air pollutants, toxic elements present in dust and their sources – collection of air samples.

Sources, effects, control of pollution and chemical analysis for the following.

(a) Primary pollutants:

- (i) Carbon compounds - Carbon monoxide(CO) and Carbon dioxide (CO₂).

(ii) Sulphur compounds- sulphur dioxide (SO₂), Sulphur trioxide (SO₃) and Hydrogen Sulphide (H₂S).

(iii) Nitrogen compounds - nitric oxide (NO), and nitrogen dioxide (NO₂),

(iv) Hydrocarbons - Aliphatic hydrocarbons and polycyclic aromatic hydrocarbons (PAH).

(v) Particulate matter-Respirable and Suspended particulate matter, Inorganic and Organic particulates.

(b) Secondary pollutants - Ozone (O₃), peroxy acetyl nitrate (PAN), peroxy benzyl nitrate (PBN)

(c) Standards for ambient air quality.

Unit- V Kinetic Methods of Analysis & Non aqueous Titrimetry

(a) Kinetic methods of analysis: introduction, slow reactions, catalyzed reactions, methods of determination of catalyst concentration, extrapolation method for the determination of catalyst, variable time method, fixed time method, examples for the determination of toxic metals and anions using some typical kinetic reactions.

(b) Non aqueous titrimetry: Classification of solvents and titrations for non-aqueous titrimetry-Types of reactions - Indicators .

(i) Determination of acids

(ii) Determination of bases

(iii) Karl-Fisher reagent for the determination of moisture content in drugs and other samples.

Text books

1. Chemical analysis – H.A Laitinan, McGraw Hill Book Co
2. Standard methods of Chemical Analysis, Welcher
2. Technical Methods of Analysis, Griffin, McGraw Hill
3. Commercial Methods of Analysis, Foster Dee Snel and Frank M. Griffin, McGraw Hill Book Co.
4. Environmental Chemistry, Anil Kumar De, Wiley Eastern Ltd.
5. Environmental Analysis, S.M Khopkar (IIT Bombay)
6. Environmental Air Analysis, Trivedi and Kudesia, Akashdeep Pub.

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SPECIALIZATION: ANALYTICAL CHEMISTRY
Paper - IV: Instrumental Methods of Analysis -II
(Effective from 2021-2022 Admitted batch)

Unit – I: Atomic Spectroscopy - I

Atomic Absorption Spectroscopy(AAS): theory-atomic line width, line broadening, sample introduction techniques-nebulization, electrothermal, **direct insertion, hydride generation laser ablation, sputtering, arc and spark ablation;** instrumentation-flame AAS and non-flame / furnace AAS, **atomization techniques:** flame, electrothermal, **glow- discharge atomization, hydride atomization, cold-vapour atomization and Inductively coupled plasma,** resonance line sources, hollow cathode lamp, sensitivity and detection limits in AAS, chemical and spectral interferences, applications with special reference to analysis of trace metals in oils, alloys and toxic metals in drinking water and effluents

Unit – II: Atomic Spectroscopy – II

(a)Flame photometry: principle of flame photometry, theory, instrumentation, combustion flames, detectors, and analysis of Na, K, Ca, Mg, Li

(b)Inductively coupled plasma Emission spectroscopy and Inductively coupled plasma Mass spectrometry (ICP-AES, ICP-MS): principle of AES, instrumentation, plasma, AES detectors, quadrupole mass spectrometers, difference between the two detectors, analysis methods for liquids and solids, applications in the analysis of trace and toxic metals in water, geological and industrial samples.

Unit – III Thermal methods of Analysis

(a) Thermo gravimetry-theory, instrumentation, applications with special reference to

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{CaC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, CaCO_3 , $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$

(b) Differential thermal analysis-principle, instrumentation, difference between TG and DTA - applications with special reference to the clays and minerals, coals (fuels)

(c)Differential scanning calorimetry-principle, instrumentation, applications to inorganic materials like chlorates and per chlorates, ammonium nitrate, organic compounds and Drugs.

Unit- IV: Electro analytical Methods of Analysis - I

(a) Voltametry and polarographic analysis : Classification of voltametry, principle of polarography, residual current, migration current, diffusion current, half-wave potential, Ilkovic equation, instrumentation, Dropping mercury electrode (DME), advantages and disadvantages of DME, DC polarography, AC polarography, Pulse polarography-Normal pulse, triangular pulse and squarewave pulse; qualitative and quantitative analysis of inorganic ions-Cu, Bi, Pb, Cd, Zn, AC polarography, pulse polarography

(b) Anode stripping voltametry: principle, instrumentation, Hanging mercury drop electrode, application in the analysis of Pb and Cd in environmental samples, principle of cathode stripping voltametry.

(c) Coulometric analysis: principles of coulometric analysis with constant current, coulometric analysis with controlled potential, applications of coulometric methods for the analysis of cations-As (III), Fe (II) and I⁻ and S²⁻ by using I₂ liberations and Ce⁴⁺ liberation in solutions

Unit – V Electro Analytical and Radio chemical methods of analysis - II

(a) Ion Selective Electrodes: reference electrodes - hydrogen electrode, calomel electrode, silver chloride electrode; indicator electrodes – hydrogen and glass electrodes, theory of membrane potentials and liquid junction potentials, types of ion selective electrodes, basic properties, potentials and construction, calibration of ion selective electrodes, ion selective electrodes with fixed membrane sites, silver, lead, cadmium, sulfide, fluoride, cyanide and glass electrodes, applications in the analysis of air and water pollutants, principles of liquid membrane, gas sensing and enzyme based electrode

(b) Radio chemical methods of analysis: detection and measurement of radioactivity, introduction to radioactive tracers, applications of tracer technique, isotope dilution analysis - applications, activation analysis – application, advantages and disadvantages, radio carbon dating technique

Text Books:

1. Instrumental methods of analysis – H.H Willard, Meritt Jr. and J.A Dean
2. Principles of instrumental analysis – Skoog and West
3. Vogels Textbook of Quantitative Inorganic analysis – J. Basset, R.C Denney, G.H Jefferey and J.Madhan
4. Instrumental methods of analysis – B.K Sarma, Goel Publishing House, Meerut
5. Instrumental methods of Analysis – Chatwal and Anand
6. Instrumental methods of Analysis – Ewing

Reference Books:

1. W. Wendtlandt, Thermal Analysis, John Wiley Sons, New York.

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ANALYTICAL CHEMISTRY
ANALYTICAL CHEMISTRY PRACTICAL – II

(With effect from 2021-2022 admitted batch)

CLASSICAL METHODS OF ANALYSIS-2

1. Water analysis
 - (i) Determination of alkalinity (CO_3^{2-} , HCO_3^-) of water samples.
 - (ii) Determination of chemical oxygen demand (COD) of drinking water and sewage water
 - (iii) Determination of biological oxygen demand (BOD) of drinking water and sewage water

2. Redox titrations
 - (i) Determination of oxalate in kidney stones by permanganometric titration.
 - (ii) Determination of Fe(II) present in an Iron tablet using KMnO_4

3. Fertilizer analysis
 - (i) Determination of nitrate from fertilizer
 - (ii) Determination of sulfur (as sulfate) from sulfur containing fertilizer.

4. Analysis of oils and soaps
 - (i) Determination of saponification value, acid value and iodine value of oil sample
 - (ii) Determination of moisture content and total alkali of soaps

5. Separation and determination of ions by ion-exchanger resins
 - (i) Determination of Na^+ by cation exchanger resin
 - (ii) Determination of Na^+ and K^+ in a mixture by cation exchanger resin
 - (iii) Determination of Cl^- and Br^- in a mixture by anion exchanger resin

INSTRUMENTAL METHODS OF ANALYSIS-2

1. pH metry

- (i) Determination of purity of commercial H_3PO_4 by pH metric titration
- (ii) Determination of CH_3COOH by pH metric titration.
- (iii) Determination of stability constant of copper glycinate

2. Conductometry

- (i) Determination of a mixture of strong acid and weak acid present in a coloured effluent
- (ii) Determination of sulphate using strong base

3. Potentiometry

- (i) Determination of Fe(II) using Mn(VII) of by potentiometric titration
- (ii) Determination of Fe (II) using V(V) of by potentiometric titration
- (iii) Determination of a mixture of Mn(VII) and V(V) with Fe(II) using potentiometric end point
- (iv) Determination of a mixture of bromide and chloride with AgNO_3 using potentiometric end point

4. Spectrophotometry

- (i) Determination of nitrite in drinking water samples by diazotization method
- (ii) Determination of nitrate -phenoldisulphonic acid method
- (iii) Simultaneous Determination of Cr(VI) and Mn(VII) in a mixture without separation
- (iv) Determination of Cu(II) using EDTA – Photometric titration method.

5. Flame photometry

- (i) Determination of Lithium by flame photometry
- (ii) Determination of calcium from milk samples using flame photometry

6. Thin layer chromatography

- (i) Separation and identification of the given mixture of colourless compounds (Diphenylamine, Benzophenone and Naphthalene)
- (ii) Separation and identification of the given mixture of coloured compounds (azobenzene, hydroxyazobenzene, p-aminoazobenzene).

References:

1. A Text Book of Quantitative Inorganic Analysis (3rd Edition) – A. I. Vogel