

Shiksha Mandal's Bajaj College of Science, Wardha

Department of Chemistry

Revised Syllabus of M. Sc. Chemistry Semester II for Academic Autonomy

(Approved in BOS Meeting 8-September-2021)

Bajaj College of Science, Wardha (Autonomous College) SUBJECT: CHEMISTRY Syllabus of M.Sc. I Semester II

Paper V [Code: PG- CHE (02)-S2-T1]

Inorganic Chemistry

60 h (4 h per week): 15 h per unit [L-T-P = 4-0-0]

80 Marks

Course Objectives

- 1. Analyse the reactivity of transition metal complexes.
- 2. Analyse the reaction pathways of complex formation understand its reaction mechanism.
- 3. Study the preparation, structures and properties of metal carbonyls and metal nitrosyls and the chemistry of dioxygen and dinitrogen complexes.
- 4. Understand the classification of metal clusters containing metal-metal bonds.
- 5. Learn about the isopolyacids and heteropolyacids and their structures.

Course Outcomes

The learner will gain knowledge of:

- 1. Reaction mechanism governing the substitution reaction substitution and electron transfer reactions in transition metal complexes.
- 2. Structure and bonding in metal carbonyls and metal nitrosyls.
- 3. Occurrence of metal-metal bonds in metal clusters and its significance.

Contents:-

Unit-I

A. Metal – Ligand Equilibria in Solution:

Stepwise and overall formation constants; trends in stepwise formation constants; factors affecting stability of metal complexes with reference to nature of metal ion, ligand, chelate effect and thermodyanamic origin. Determination of formation constant by: (1) spectrophotometric method (Job's and Mole ratio method) (2) Potentiometric method (Irving - Rossotti Method)

B. Reaction mechanism of transition metal complexes-I:

Energy profile of reaction, reactivity of metal complexes, inert and labile complexes, kinetics of octahedral substitution: Acid hydrolysis, various types of acid hydrolysis, factors affecting acid hydrolysis, stereochemistry of intermediates in SN¹ and SN², Base hydrolysis, Conjugate base (SN¹CB) mechanism, Essential requirements for SN¹CB mechanism, Direct and indirect evidences in favor of conjugate mechanism, anation reaction, reaction without metal ligand bond breaking.

Unit-II

Reaction mechanism of transition metal complexes -II:

Substitution reaction in square planar complexes: trans effect, cis effect, steric effect, solvent effect, effect of leaving group, effect of charge, effect of nucleophile, effect of temperature. Trans effect theories (Electrostatic polarization theory; pi bonding theory), use of trans effect, mechanism of substitution reaction of Pt(II) Complexes, electron transfer reactions. Types of electron transfer

15h

reaction, conditions of electron transfer and mechanism of one electron transfer reaction, outer sphere and inner sphere mechanism, two electron transfer reactions, complementary and non-complementary reaction, tunneling effect, cross reactions.

Unit-III: Metal π-Complexes:

A. Metal carbonyls

Structure and bonding, vibrational spectra of metal carbonyls for bonding and structure elucidation, important reaction of metal carbonyls. Metal carbonyl clusters with reference to classification, EAN rule, 18 electron rule, synthesis and structures.

B. Metal nitrosyls

Nitrosylating agents for synthesis of metal nitrosyls, EAN rule. vibrational spectra and X-ray diffraction studies of transition metal nitrosyls for bonding and structure elucidation, important reactions of transition metal nitrosyls, structure and bonding. Application of Dinitrogen and dioxygen complexes. Application of Wilkinson's catalyst and Vaska's compound.

Unit-IV: Cluster II

Metal-Metal Bond:

Occurrence of metal-metal bonds, Classification of metal cluster: binuclear, trinuclear, tetranuclear, pentanuclear and hexanuclear with reference to halide, oxide, alkoxide and acetate clusters. Isopoly, heteropoly acids and their anions.

Reference books:

1] S. F. A. Kettle, J. N. Murral & S. T. Teddler: Valency Theory

- 2] C. A. Coulson: Valency
- 3] J. E. Huheey: Inorganic Chemistry
- 4] F .A. Cotton & G. Wilkinson: Advanced Inorganic Chemistry 3rd , 5th & 6th Editions.
- 5] A. F. Willims: Theoretical Approach in inorganic chemistry.
- 6] A. Mannas Chanda: Atomic Structure and chemical Bonding
- 7] L. E. Orgel: An introduction to transition metal chemistry, Ligand field theory, 2nd Edition.
- 8] J. J. Logowski: Modern Inorganic Chemistry
- 9] B. Durrant and P. J. Durrant: Advanced Inorganic Chemistry
- 10] J. C. Bailar: Chemistry of co-ordination compounds.
- 11] W. L. Jolly: Modern Inorganic Chemistry
- 12] R. S. Drago: Physical methods in inorganic chemistry.
- 13] Waddington: Nonaqueous solvents.
- 14] Sisler: Chemistry of non-aqueous solvents.
- 15] A. K. Barnard: Theoretical Inorganic Chemistry
- 16] Emeleus and Sharpe: Modern Aspect of Inorganic Chemistry.
- 17] F. A. Cotton: Chemical Applications of Group theory.
- 18] Jones: Elementary Co-ordination chemistry.
- 19] B. N. Figgis: Introduction to Ligand field.
- 20] S. F. A. Kettle: Co-ordination chemistry.
- 21] M. C. Day and J. Selbin: Theoretical Inorganic Chemistry.
- 22] J. Lewin and Wilkins: Modern Co-ordination chemistry.
- 23] Gowarikar, Vishwanathan and Sheedar: Polymer science.
- 24] R. L. Dutta and A. Symal: Elements of magneto chemistry
- 25] P. Atkins: Inorganic Chemistry 4th Edition, Oxford University Press.
- 26] D. M.P. Mingos: Essential Trends in Inorganic Chemistry, Oxford University Press
- 27] Bertini, et al: Bioinorganic Chemistry (Viva)

15h

- 28] Fenton, David E.: Bio coordination chemistry, Oxford
- 29] Selected Topics in Inorganic Chemistry by Wahid U Malik, Tuli, Madan.
- 30] A Logical Approach to Modern Inorganic Chemistry by Jagdamba Singh
- 31] Essentials of Bio Inorganic Chemistry by Monal Singh, Neerja Gupta
- 32] Concise Coordination Chemistry by R. Gopalan , V. Ramalingam
- 33] Advanced inorganic chemistry Volume I by Madan, Malik, Tuli Prakash S. Chand publication
- 34] Advanced inorganic chemistry Volume II Madan, Malik, Tuli Prakash S. Chand publication

Semester II Paper VI [Code: PG- CHE (02)- S2-T2] Organic Chemistry

60 h (4 h per week): 15 h per unit

80 Marks

Course Objectives

To learn

- 1. various addition reactions across C-C, C-O and C-N multiple bonds.
- 2. basic concepts in molecular rearrangement & elimination reaction.
- 3. types and mechanism of free radical reactions.
- 4. principles and advantages of green chemistry.

Course Outcomes

Students will gain an understanding of:

- 1. various addition reactions across C-C, C-O and C-N multiple bonds.
- 2. basic concepts in molecular rearrangement & elimination reaction.
- 3. types and mechanism of free radical reactions.
- 4. principles and advantages of green chemistry.

Contents:-

Unit-I

Addition reactions:

A. Addition to carbon-carbon multiple bond: Mechanistic and stereochemical aspects of addition reaction involving electrophiles, nucleophiles and free radicals, regio and chemoselectivity, Orientation and stereochemistry, Addition to cyclopropanes, Hydrogenation of double bond and triple bonds. Hydrogenation of aromatic rings, hydroboration..

B. Addition to carbon-hetero atom multiple bond: Mechanism of metal hydride reduction of saturated and unsaturated carbonyl compounds, acids, esters, and nitriles, Addition of Grignard reagents, organozinc and organolithium reagents to carbonyls and unsaturated carbonyl compounds, Wittig reaction, Mechanisms of condensation reactions involving enolates- Aldol, Knoevengel, Claisen, Mannich, Benzoin, Perkin, Stobbe reaction, Michael reaction and Robinson annulations. Hydrolysis of esters and amide

Unit-II

Molecular rearrangements:

A. Molecular rearrangements: Classification and General mechanistic treatment of electrophilic, nucleophilic and free radical moleculer rearrangement. Mechanism and synthetic applications of Wagner-Meerwin, Pinacol-Pinacolone, Tiffenev-Demjnov ring expansion, benzil-benzilic acid, Favorski, Baeyer Villiger, Wolff, Arndt-Eistert synthesis, Curtius Lossen, Beckman, Hoffman, Schmidt rearrangement.

B. Elimination reactions: E1, E2, E1CB mechanisms, orientation and stereochemistry in elimination reaction, Saytzeff and Hoffman's rule, Effect of substrate structure, attacking base, leaving group and medium, competition between elimination and substitution, syn eliminations, pyrolytic elimination.

15h

Unit-III

Free radical reactions:

Generation of free radicals, types and mechanism of free radical reactions, free radical substitution mechanism at an aromatic substrate, aliphatic substrate, reactivity at a bridgehead position, Neighbouring group assistance, reactivity for aliphatic and aromatic substrates, reactivity in attacking radicals, effect of solvent on reactivity, Halogenation at an alkyl carbon, allylic carbon (NBS), hydroxylation at an aromatic carbon by means of Fenton's reagent. Auto-oxidation, chlorosulphonation (Reed Reaction) Coupling of alkynes and arylation of aromatic compounds by diazonium salts, Sandmeyer reaction, Free radical rearrangement: Hunsdiecker reaction, Iododecarboxylation, Barton reaction, Hoffmann-Loefler-Freytag reaction.

Unit-IV

Green chemistry:

Twelve basic principles of green chemistry, calculation of atom economy of rearrangements, addition, substitution and elimination reaction with suitable examples, Case study of Bhopal gas tragedy and Seveso disaster, Synthesis involving basic principles of green chemistry- paracetamol, Ibuprofen, hydroquinone, adipic acid, ε -caprolactum, styrene, urethanes, Free radical bromination, Multi-component reactions (Biginelli, Ugi and Passerini reaction), Prevention or minimization of hazardous products, choice of solvents. Sonochemistry, microvave induced reactions, polymer supported reagents, reactions in aqueous medium, zeolites and ionic liquid supported reaction, Solvent free reactions, electrochemical reactions, Biocatalysts in Organic synthesis.

Reference books:

- 1) Advanced Organic Chemistry -Reaction mechanism and structure. Jerry March, John Wiley
- 2) Advanced Organic Chemistry- Part-A- F.A. Carey and R. J. Sunberg, Plenum
- 3) A Guidebook to Mechanism in Organic Chemistry-Peter Skyes, Longman
- 4) Structure and Mechanism in Organic Chemistry-C.K. Gold, Cornell University Press
- 5) Organic Chemistry, R.T. Morrison Boyd. Prentice Hall
- 6) Modern Organic Chemistry-H.O. House, Benjamin

7) Principal of Organic Chemistry-R.O.C. Norman and J.M. Coxon, Blackie Academic and Professional

- 8) Reaction Mechanism in Organic Chemistry-S.M. Mukharji and S.P. Singh, Macmilan
- 9) Advanced Organic Chemistry L. F. Fieser and M. Fieser.
- 10) Organic Chemistry Vol. I and II I. L. Finar
- 11) Frontier Orbitals and Organic Chemical Reactions-I. Fleming
- 12) Orbital Symmetry R. E. Lehr and A. P. Marchand
- 13) Reactive Intermediate in Organic Chemistry-N. S. Isaacs
- 14) A Textbook of organic chemistry- R.K. Bansal
- 15) Some Modern Methods of Organic Synthesis-W. Carruthers
- 16) Advance Organic Chemistry Part-B-F. A. Caray and R. J. Sundberg Plenum Press
- 17) Modern Synthetic Reaction. H. O. House and W. A. Benjamin
- 18) Designing Organic Synthesis-S. Warren
- 20) Organic Reaction and their Mechanism-P. S. Kalsi
- 21) New trends in green chemistry -V.K. Ahluwalia and M. Kidwai, Anamaya publishers New Delhi

Semester II Paper VII [Code: PG- CHE (02) - S2-T3] Physical Chemistry

60 h (4 h per week): 15 h per unit [L-T-P = 4-0-0]

80 Marks

Course Objectives

- 1. To recapitulate fundamentals of quantum chemistry and extending the study to derivations of wave functions for particle in three dimensional box and hybrid orbitals.
- 2. To study various electrochemical processes, thermodynamic quantities, potentials and different types of fuel cells.
- 3. To revise the fundamentals of solid state and understand the nature as well thermodynamics of crystal defects and nonstoichiometry.
- 4. To get introduced to fundamentals of Statistical Thermodynamics.
- 5. To study important adsorption isotherms as well as important aspects of micelles.

Course Outcomes

On successful completion of this course the students should be able to-

- 1. Derive the wave functions for particle in three dimensional box and hybrid orbitals: understand the concept of degeneracy and solve related numericals.
- 2. Understand faradic-nonfaradic processes, thermodynamics of cell, electrochemical potentials and some important fuel cells.
- 3. Learn the basics of solid state chemistry and gain some understanding of crystal defects and nonstoichiometry.
- 4. Know the important concepts of statistical thermodynamics with preliminary idea of Maxwell Boltzmann, Bose Einestein, Fermi Dirac statistics.
- 5. Derive important adsorption isotherms as well as realize some important aspects of micelles.

Contents:-

Unit-I

Quantum Chemistry:

Recapitulation, Postulates of Quantum Mechanics, Operator algebra, Commutator of operators and Numericals. Eigen values, Eigen functions and Numericals. Normalized, orthogonal wave functions and Numericals. Particle in three dimensional box, degeneracy of energy levels, Quantum mechanical principles of Hybridization, Construction of wave functions for sp, sp² and sp³ hybrid orbitals. Treatment of linear harmonic oscillator and rigid rotor. Approximate methods. Solving Electronic Schrödinger equation for Hydrogen atom

Unit-II

Electrochemistry

Introduction and over view of Electrochemical Processes, Electrochemical Cell and Reactions, Faradic and Nonfaradiac Processes, Basic Electrochemical Thermodynamics, Free Energy and cell EMF, Half Reaction and Reduction Potentials, Formal Potentials, Reference Electrodes, Measurements of Potential Differences, Electrochemical Potentials, Fermi Level and Absolute Potentials, Liquid Junction Potential. Electrochemical devices: Alkaline fuel cells, Phosphoric acid fuel cells, High temperature fuel cells.

15h

Unit-III

Solid State Chemistry:

A. Introduction: Introduction to crystals, Unit Cell and lattice parameters, Symmetry elements in crystals, Absence of fivefold axis, Space groups, The Bravais Lattices, Miller Indices, Bragg's Equation, seven crystal system, Packing in crystals, Hexagonal Closest Packing (HCP) Cubic Closest Packing (CCP), Voids, packing fraction, Numericals.

B. Crystal Defects and Non-stiochiometry: Perfect and imperfect crystals, point defects, line and plane defects. Thermodynamics of Schottky and Frenkel defect formation, colour centers, non-stiochiometry and defects. Numericals.

Unit-IV

Statistical Thermodynamics and Surface Chemistry:

- A. Statistical thermodynamics: Lagrange's Method of Undetermined Multipliers (Conditional Maximization), Stirling Approximation, Concept of Distribution, Thermodynamic Probability and most probable distribution, Maxwell Boltzmann, Bose Einestein, Fermi Dirac statistics, comparison between three statistics.
- **B.** Surface Chemistry: Adsorption definition, Thermodynamics of adsorption, Langmuir adsorption isotherm, Langmuir constant and Gibbs energy of adsorption, Langmuir adsorption with lateral interaction, BET adsorption isotherm, adsorption on heterogeneous surface, the potential theory of Polanyi.
- **C. Micelles:** Micellization, Hydrophobic Interaction, Critical Micellar Concentration (CMC), factors affecting CMC of surfactants, thermodynamics of micellzation mass action models, micellar solubilization, Numericals.

Reference books:

- 1] Ira .N. Levine, Quantum Chemistry, 5th edition (2000), Pearson educ., Inc.New Delhi
- 2] A.K.Chandra, Introductory Quantum Chemistry, 4th edition (1994), Tata Mcgraw Hill, New Delhi.
- 3] M.W.Hanna, "Quantum Mechanics in Chemistry", Benjamin
- 4] L. Pualing and E. B. Wilson, Introduction to Quantum Mechanics with Applications to Chemistry, McGraw Hill, New York (1935).
- 5] R. K. Prasad, Quantum Chemistry, New Age International, Delhi.
- 6] R. K. Prasad, Quantum Chemistry through problems and solutions, New Age International, New Delhi, 2009.
- 7] B. C. Reed, Quantum Mechanics, Jones and Bartlett, New Delhi, 2010.
- 8] S. Glasstone, An Introduction to Electrochemistry, East-West Press Pvt. Ltd., New Delhi, 2004.
- 9] H. K. Moudgil, Text Book of Physical Chemistry, Pretice Hall of India, New Delhi, 2010.
- 10] S. O. Pillai, Solid State Physics, New Age International, New Delhi, 2102.
- 11] N. B. Hanny, Treaties in Solid State Chemistry,
- 12] M. C. Day and J Selbin, Theoretical Inorganic Chemistry, Reinhold Pub. Corp., New York,
- 13] M. C. Gupta, Statistical Thermodynamics, New Age International.
- 14] K. Huang, Statistical Mechanics, Wiley, New Delhi, 2003.
- 15] Andrew Maczek, Statistical Thermodynamics, Oxford University Press Inc., New York (1998).
- 16] C.Kittel, "Introduction to solid state Physics", Wiley
- 17] L.V.Azaroff, "Introduction to solids", McGraw Hill
- 18] L. E. Smart and E. A. Moore, Solid State Chemistry-An Introduction, CRC Tylor and Fransis, 2005.
- 19] C. N. R. Rao and Gopalakrishnan, "New Directions in Solid State Chemistry" Second Edition, Cambridge University Press.
- 20] Anthony R. West, "Solid State Chemistry and its Applications" Wiley India Edition

- 21] D. K. Chakravorty, Solid State, New Age International.
- 22] Modern Electrochemistry, Volume 1 and 2, J.O.M Bokris and A.K.N, Reddy Plenum Press N.Y. (1970)
- 23] Electrochemical Methods second edition, A.J. Bard and L.R. Faulkner, John Wiley and Son (2001).
- 24. Physics and Chemistry of Interface, second edition, Hans-Jurgen Butt, Karlheunz Graf, Micael Kappl, Willey VCH (2006), ISBN-13 978-3-527-40629-6
- 25] Physical Chemistry of Surface, A.W. Admson, fifth edition, Wiley Interscience Publiation (1990)
- 26] Surfactant Science and Technology, second edition, Drew Myers, VCH Publishers (1992)
- 27] Principles of Colloids and Surface Chemistry, P.C. Hiemenz Marcel and Dekker, N.Y. (1977)

e-References:-

e-PGPathshala (inflibnet.ac.in)

Semester II Paper VIII [Code: PG- CHE (02)- S2-T4] Analytical Chemistry

60 h (4 h per week): 15 h per unit [L-T-P = 4-0-0]

Course Objectives

The aim of this course is to provide students with a broad understanding of the principles and applications of analytical chemistry.

1. Students are first provided with an introduction into sample collection and preparation.

This course will cover advanced separation techniques:

2. Gas chromatography, HPLC & Super critical fluid Chromatography.

Finally, students will also be introduced to basic and commonly used instrumental methods of chemical analysis:

- 3. atomic absorption spectroscopy
- 4. some electroanalytical methods.

Course Outcomes

After the successful completion of this course, students will -

- 1. Be able to plan for sampling and understand how different sampling methods can be used in speciation studies.
- 2. Be familiar with Stoichiometric and sub-stoichiometric reactions and calculations.
- 3. Understand the theoretical principles of modern separation techniques Gas chromatography & HPLC.
- 4. Get introduced to new separation method Supercritical fluid chromatography.
- 5. Get knowledge of the theoretical principles of modern analytical techniques AAS.
- 6. Realize the theoretical principles of electroanalytical techniques Polarography & Ampereometry.

Unit-I

Sampling and Quantification:

A. Sampling and sample treatment: Criteria for representative sample. Techniques of sampling of gases (ambient air and exhaust gases), liquids (water and milk samples), solids (soil and coal samples) and particulates. Hazards in sampling. Safety aspects in handling hazardous chemicals. Sample dissolution methods for elemental analysis: Dry and wet ashing, acid digestion, fusion processes and dissolution of organic samples.

B. Detection and quantification: Concepts and difference between sensitivity, limit of detection and limit of quantification, role of noise in determination of detection limit of analytical techniques. Units in chemical analysis and their interconversion.

Unit-II

Modern separation techniques:

A. Gas Chromatography: Principle including concept of theoretical plates and van-Deemter equation. Instrumental set up- carrier gas, sampling system, column and detector. Types of columns, their advantages and limitations. Detectors in GC analysis. Temperature programmed GC. Factors affecting retention, peak resolution and peak broadening.

B. Liquid chromatography: Principle, Instrumentation, Advantages and applications of HPLC. Types of columns and detectors. Principle and applications of size exclusion, gel permeation, ion retardation, normal phase and reverse phase chromatography.

80 Marks

15h

C. Supercritical fluid chromatography: Introduction and applications.

Unit-III

Optical methods of analysis-II:

- **A. Atomic absorption spectroscopy:** Principle. Atomic energy levels. Grotrian diagrams. Population of energy levels. Instrumentation. Sources: Hollow cathode lamp and electrodeless discharge lamp, factors affecting spectral width. Atomizers: Flame atomizers, graphite rod and graphite furnace. Cold vapour and hydride generation techniques. Factors affecting atomization efficiency, flame profile. Monochromators and detectors. Beam modulation. Detection limit and sensitivity. Interferences and their removal. Comparison of AAS and flame emission spectrometry. Applications of AAS.
- **B.** Inductively coupled plasma-atomic emission spectroscopy: Principle, atomization and excitation. Plasma source and sample introduction. Instrumentation. Comparison of Flame photometry with ICP-AES & AAS. Applications.

Unit-IV

Electrochemical methods of analysis-II:

A. Polarography: Principle of DC polarography. Instrumentation in polarography. Advantages and limitations of DME. Types of currents- residual current, migration current, diffusion current, limiting current, adsorption current, kinetic current and catalytic current. Ilkovic equation-diffusion current constant and capillary characteristics. Derivation of equation of polarographic wave and half wave potential. Experimental determination of half wave potential. Reversible, quasi reversible and irreversible electrode reactions. Polarographic maxima and maximum suppressor. Oxygen interference and deaeration. Introduction to pulse, a.c. and oscillographic techniques and their advantages. Applications of polarography in determination of dissolved oxygen, metal ion quantification and speciation, simultaneous determination of metal ions, analysis of organic compounds. Limitations of polarography.

B. Amperometric titrations: Principle, types and applications in analytical chemistry.

<u>Reference books</u>:

- 1] Quantitative analysis: Day and Underwood (Prentice-Hall of India)
- 2] Vogel's Text Book of Quantitative Inorganic Analysis-Bassett, Denney, Jeffery and Mendham (ELBS)
- 3] Analytical Chemistry: Gary D. Christian (Wiley India).
- 4] Instrumental Methods of Analysis: Willard, Merrit, Dean, Settle (CBS Publishers, Delhi, 1986)
- 5] Sample Pre-treatment and Separation: R. Anderson (John Wiley and Sons)
- 6] Stoichiometry: B. I. Bhatt and S.M. Vora, 2nd Edition (Tata Mc-Graw Hill publication)
- 7] Instrumental Methods of Chemical Analysis: Braun (Tata McGraw-Hill)
- 8] Advanced Analytical Chemistry: Meites and Thomas (McGraw-Hill)
- 9] Instrumental Methods of Analysis: G. Chatwal and S. Anand (Himalaya Publishing House)
- 10] Analytical Chemistry: Problems and Solution, S. M. Khopkar (New Age International Publication)
- 11] Basic Concepts in Analytical Chemistry: S. M. Khopkar (New Age International Publication)
- 12] Advance Analytical Chemistry: Meites and Thomas: (Mc Graw Hill)
- 13] An Introduction to Separation Science: L. R. Shyder and C. H. Harvath (Wiley Interscience)
- 14] Fundamental of Analytical Chemistry: S. A. Skoog and D. W. West
- 15] Instrumental Methods of Chemical Analysis: G. W. Ewing
- 16] Polarography: Koltoff and Ligane
- 17] Electroanalytical Chemistry: Sane and Joshi (Quest Publications)

15h

Semester II Practical-III [Code: PG- CHE (02)- S2-P1] Organic Chemistry

12 h per week [L-T-P = 0-0-8]

Course Objectives

To learn the purification techniques (solvents & reagents), qualitative analysis of two component mixture & organic preparation of single step & two step synthesis.

Course Outcomes

Students will gain an understanding of:

- 1) Separation and analyze the different component mixtures of simple organic compounds.
- 2) Purification, Crystallization, and different Distillation processes.
- 3) How to calculate a limiting reagent, yield, and percent yield
- 4) Synthesis using substitution and condensation reactions.
- 5) Single step & multistep synthesis.

Contents:-

I. Purification techniques (Demonstrations):

a) Purification of solvents and reagents using techniques like crystallization, distillation, steam distillation, vacuum distillation etc.

- b) Chromatography: TLC, Column, paper
- c) Solvent extraction using soxhlet extractor

II. Qualitative Analysis:

Two component mixture separation using chemical and physical techniques and using solvents. (8 mixtures minimum)

III. Organic Preparations (minimum 8 preparations):

Spectral characterization of prepared compounds wherever possible:

1. Single step preparation

- a) Aldol condensation: Benzaldehyde \rightarrow Dibenzal acetone (chalcone)
- b) Benzophenone \rightarrow benzhydrol
- c) Nitrobenzene \rightarrow m-di-nitrobenzene
- d) m-di-nitrobenzene \rightarrow m-nitroaniline
- e) Methyl acetoacetate \rightarrow 5-methyl-isoxazol-3-ol
- f) Ethyl acetoacetate \rightarrow 4-aryl-6-methyl-3,4-dihydro-2(1H)-pyrimidinone ester
- g) Ethyl acetoacetate → Diethyl 1,4-dihydro-2,6-dimethyl-4-phenylpyridine-3,-5dicarboxylate
- h) Sulphanilic acid \rightarrow Methyl orange

i) p-nitroaniline \rightarrow p-red

- 2. Two step preparation
- a) Acetanilide \rightarrow p-nitroacetanilide \rightarrow p-nitroaniline
- b) Aniline $\rightarrow 2,4,6$ -tribromo aniline $\rightarrow 2,4,6$ -tribromo acetanilide
- c) Nitrobenzene \rightarrow m-dinitrobenzene \rightarrow m-nitroaniline
- d) benzophenone \rightarrow benzophenoneoxime \rightarrow Benzanilide
- e) Chlorobenzene \rightarrow 2,4-dinitrochlorobenzene \rightarrow 2,4-dinitrophenylhydrazine
- f) Glycine \rightarrow Benzoyl glycine(hippuric acid) \rightarrow 4-benzilidene-2-phenyl oxazole

100 Marks

Reference Books:-

- 1. A Textbook of Practical Organic Chemistry, 4th Edn., A. I. Vogel, ELBS.
- 2. Laboratory Techniques in Organic Chemistry, V. K. Ahluwalia, Pooja Bhagat, Renu Agrawal, I. K. International
- 3. Practical Organic Chemistry: Qualitative Analysis, Ane's Student Edition, S. P. Bhutani, Aruna Chhikara, Ane Books India
- 4. Advanced Practical Organic Chemistry, John Leonard, Barry Lygo, Garry procter, CRC Press, Special Indian Edition
- 5. Organic Chemistry A Lab Manual, Pavia, Lampman, Kriz, Engel, Cengage learning
- 6. Practical Organic Chemistry, F. G. Mann and B. C. Saunders, English language Book Society
- 7. Organic Chemistry: Laboratory Course book, Dr. P. V. Tekade, Selective and Scientific books, New Delhi

Semester II Practical-IV [Code: PG- CHE (02)- S2-P2] Analytical Chemistry

12 h per week [L-T-P = 0-0-8]

100 Marks

Course Objectives

Analytical measurements are aimed at obtaining qualitative and quantitative information about the composition and structure of various materials that have relevance to both fundamental understanding as well as applications towards improving the quality of life. Here students develop an understanding of the broad role of the chemist in measurement and problem solving for analytical tasks. Also experience some scientific methods employed in analytical chemistry.

Course Outcomes

- 1. Performing classical analytical experiments, and make observations and assessments of important factors that could affect the analytical result.
- 2. Use of digital tools MS-Excel to present chemical analytical work.
- 3. Understanding of methods for calibration and sampling applied to quantitative analysis.
- 4. Understanding the application of analytical methods based on titrations, separations, electrochemical measurements, and spectroscopy at an introductory level.

Contents:-

Section (A):

I. Classical methods and separation techniques: Calibration, validation and computers

- 1) Calibration of pipette and burette.
- 2) Statistical analysis of data.
- 3) Use of MS-Excel in statistical analysis of data and curve fitting.

II. Volumetry

- 1) Determination of Na₂CO₃ in washing soda.
- 2) Determination of NaOH and Na₂CO₃ in a mixture.
- 3) Estimation of nickel in given solution by direct complexometric titration with EDTA using bromopyrogallol red.
- 4) Estimation of nickel in given solution by complexometric back-titration with EDTA.
- 5) Estimation of chloride in given solution by Mohr's titration.
- 6) Estimation of chloride in given solution by Volhard's titration.
- 7) Determination of volume strength of commercial hydrogen peroxide by redox titration with KMnO₄.
- 8) Estimation of phenol/ aniline by bromination method.
- 9) Estimation of glucose.
- 10) Estimation of acetone.
- 11) Estimation of formaldehyde.
- 12) Estimation of Mn in the presence of Fe using masking phenomenon (ferromanganese alloy).

III. Gravimetry

1) Estimation of barium as barium sulphate.

2) Estimation of calcium as calcium oxalate/ calcium carbonate/ calcium oxide.

IV. Separation techniques

- 1) Qualitative separation of metal ions by paper chromatography for 2/3 components.
- 2) Determination of ion-exchange capacity of resin.
- 3) Separation of ions by ion exchange.

Section (B): Instrumental techniques

I. Electroanalytical techniques

- 1) Analysis of commercial vinegar by conductometric titration.
- 2) Estimation of phenol by conductometric titration with NaOH.
- 3) Determination of strength of HCl and CH₃COOH in a mixture conductometrically.
- 4) Determination of strength of HCl and oxalic acid in a mixture conductometrically.
- 5) Determination of strength of oxalic acid and CH₃COOH in a mixture conductometrically.
- 6) Determination of degree of dissociation and dissociation constant of acetic acid conductometrically.
- 7) Estimation of phenol in dilute solution by conductometric titration with NaOH.
- 8) Determination of strength of HCl and CH₃COOH individually and in a mixture potentiometrically.
- 9) Determination of Fe(II) by potentiometric titration with $K_2Cr_2O_7$.
- 10) Determination of three dissociation constants of H₃PO₄ by pH-metric/potentiometric titration

II. Optical methods

- 1) Determination of pK of indicator by colorimetry.
- 2) To estimate the amount of NH₄Cl colorimetrically using Nesseler's Reagent.
- 3) To study the complex formation between Fe(III) and salicylic acid and find the formula and stability constant of the complex colorimetrically (Job's method).
- 4) To determine the dissociation constant of phenolphthalein colorimetrically.

5) Estimation of iron in wastewater sample using 1,10-phenanthroline.

(Note: One experiment from each section should be performed in the examination.)

Reference Books:-

- Svehla, G. Vogel's Qualitative Inorganic Analysis, Pearson Education, 2012.
- Mendham, J. Vogel's Quantitative Chemical Analysis, Pearson, 2009.
- Willard, H. H., Instrumental methods of analysis, Wiley, 1988
- Khosla, B. D.; Garg, V. C. & Gulati, A. *Senior Practical Physical Chemistry*, R. Chand & Co.: New Delhi (2011)
- Das R. C., Behra B., Experimental Physical Chemistry, Tata McGraw Hill.
- Yadav J. B., Advanced Practical Physical Chemistry, Goel Publishing House.
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- <u>http://nsdl.niscair.res.in</u>
- <u>http://ocw.mit.edu</u>

IV. Separation techniques

- 1) Qualitative separation of metal ions by paper chromatography for components.
- Determination of ion-exchange capacity of resin.
- Separation of ions by ion exchange.

Section (B): Instrumental techniques

1. Electroanalytical techniques

- 1) Analysis of commercial vinegar by conductometric titration.
- Estimation of phenol by conductometric titration with NaOH.
- 3) Determination of strength of HCI and CH3COOH in a mixture officiality.
- 4) Determination of strength of HCl and oxalic acid in a mixture conductometrically.
- 5) Determination of strength of oxalic acid and CH3COOH in a mixture conductometrically.
- 6) Determination of degree of dissociation and dissociation constant of acetic acid conductometrically.
- 7) Estimation of phenol in dilute solution by conductometric fitration with NaOH.
- 8) Determination of strength of HCI and CH3COOH individually and, in a mixture potentiometrically.
- 9) Determination of Fe(II) by potentiometric titration with K2Cr2O7.
- 10) Determination of three dissociation constants of H₃PO₄ by pH-metric/potentiometric titration

II. Optical methods

- 1) Determination of pK of indicator by colorimetry.
- 2) To estimate the amount of NH4Cl colorimetrically using Nesseler's Reagent.
- 3) To study the complex formation between Fe(III) and salicylic acid and find the formula and stability constant of the complex colorimetrically (Job's method).
- 4) To determine the dissociation constant of phenolphthalein colorimetrically.
- 5) Estimation of iron in wastewater sample using 1,10-phenanthroline.

(Note: One experiment from each section should be performed in the examination.)

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- Mendham, J. Vogel's Quantitative Chemical Analysis, Pearson, 2009.
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