ST. JOSEPH'S COLLEGE (AUTONOMOUS)

BENGALURU 560 027

Course Structure and Syllabus for

Post Graduate Diploma in Chemistry for Industry

(Two Semester Course)

2021 onwards

Department of Chemistry

Preamble

Industrial sector requires chemists having specialized skills in analysis and research for their quality control and R&D units. Department of Chemistry has designed this career-oriented course, in collaboration with industry, which is aimed at providing necessary skills to make young graduates industry-ready. The course emphasizes on hands-on training in analytical methods while providing adequate theoretical knowledge in specialized areas of analytical chemistry.

Duration: 1 year (2 semesters)

Credits: 40

Eligibility: B.Sc. degree in Chemistry (main)/ Chemistry as one of the major subjects with a minimum of 55% marks for general merit and 50% for OBC/SC/ST.

Job opportunities: As quality control chemist/technical assistant/lab technician in chemical industries/pharmaceutical industries/ research laboratories.

Summary of credits

Total no. of hrs in the semester	Credit	Number of hrs per week	Title	Code number
Semester -I				
30	2	2	Chemical laboratory management and best laboratory practices-I	
30	2	2	Principles of chemical analysis-I	
30	2	2	Separation techniques	
30	2	2	Spectroscopy- I	
40	1.5	4	Practical I Introductory laboratory techniques and practices	
40	1.5	4	Practical II Volumetric and gravimetric analysis	
40	1.5	4	Practical III- Instrumental methods I- Spectroscopy	
40	1.5	4	Practical IV- Instrumental methods II- Chromatography	
	6		Industry visit and preparation of report	

Total hrs in the semester	Credit	Number of hrs per week	Title	Code number
Semester -II				
30	2	2	Chemical laboratory management and best laboratory practices- II	
30	2	2	Principles of chemical analysis- II	
30	2	2	Spectroscopy- II	
40	1.5	4	Practical V- Application of software in chemistry	
40	1.5	4	Practical VI: Instrumental methods III- Assorted techniques	
40	1.5	4	Practical VII- Instrumental methods IV- Spectroscopy	
40	1.5	4	Practical VIII- Instrumental methods V- Chromatography	
	8		Dissertation	

I SEMESTER- THEORY PAPERS

Course title	**CHEMICAL LABORATORY MANAGEMENT AND BEST LAB PRACTICES - I**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. **History and importance of lab safety** **6 h**

   Chemical industries in India, their contribution to GDP, job opportunities in industry, need for a course in PG diploma in Chemistry (not to be tested)

   Historical events, significance of lab safety and security, duties of chemist in ensuring safety, chemical hazards, risks in chemical laboratory, accounting of safety equipment, moral, legal and financial implications.

2. **Personal safety gears and safety equipment** **2 h**

   Laboratory clothing, foot protection, eye and face protection unit, safety shield, fire extinguishers, heat and smoke detector, respirators, safety showers, eye wash unit.

3. **Introduction to laboratory safety and chemical safety** **4 h**

   Importance of lab safety, steps for creating a risk-free laboratory environment, impact of laboratory safety, chemical safety and security management.

4. **Exposure to toxic chemicals** **5 h**

   Primary contact - inhalation, contact with skin and eye, medium of ingestion. Secondary contact. Assessing dangers with acute toxicology, specific chemical hazard, first aid for contact of different chemicals on skin, eyes, inhalation and ingestion.

5. **Handling and maintenance of equipment** **2 h**

   Principle, operation/maintenance of electric equipment, dealing with high pressure gas systems, managing high/low pressure and temperature conditions.

6. **Effective chemical management** **11 h**

   Planning strategy, standard protocols for working with hazardous chemicals, handling of toxic and reactive chemicals - substances of high toxicity, bio-hazardous materials, highly flammable chemicals, highly reactive or explosive chemicals. Use of inert atmosphere and dry conditions.

References

1. Chemistry Laboratory Safety and Security, A Guide Prudent Chemical Management; Lisa Moran and Tina Masciangioli; National Academic press. (2010).
2. Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards; Dr. Ralph J. Cicerone and Dr. Charles M. Vest; ISBN 978-0-309-13864-2; National Academic press.
3. Handbook- Good laboratory practices; Dr R. Ridley, Published by WHO and TDR (2001).

Course outcomes: After learning this paper students will be able to

CO1	Knowledge	List the basic safety practices in laboratory Recall first aid for chemical exposure, ~~and~~ toxic and reactive chemicals, and effective chemical management
CO2	Understand	Explain lab safety and security, dangers with chemicals, risks in chemical laboratory, legal and financial implications and operation/handling of electric/safety equipment
CO3	Apply	Apply SOP for handling hazardous chemicals Develop standard protocols for working with hazardous chemicals
CO4	Analyze	Inspect the toxicity of chemicals, assessing dangers with acute toxicology
CO5	Evaluate	Assess the risks involved in a chemical laboratory
CO6	Create	Design a risk -free laboratory environment

Blue print: Paper code:

Chapter number	**Title**	**Number of teaching hours (as given in syllabus)**	**Maximum marks for which questions are to be framed (including extra questions)**
1	History and importance of lab safety	6	10
2	Personal safety gears and safety equipment	2	3
3	Introduction to laboratory safety and chemical safety	4	7
4	Exposure to toxic chemicals	5	9
5	Handling and maintenance of equipment	2	3
6	Effective chemical management	11	18
Total marks excluding extra questions			35
Total marks including extra questions			50

Course title	**PRINCIPLES OF CHEMICAL ANALYSIS- I**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. Errors in chemical analysis, statistical data treatment and evaluation

16 h

Significant Figures: Least count. Rounding off numbers. Addition and subtraction; multiplication and division.

Errors**:** Some important terms replicate, outlier, accuracy and precision. Errors affecting precision and accuracy; systematic errors: sources and types of systematic errors with examples. Ways of expressing accuracy: absolute and relative errors; constant and proportional errors. Detection of systematic instrument and personal errors. Identification and compensation of systematic method errors. Terms used to describe precision of a set of replicate measurements. Mean and median. Deviation and average deviation from the mean.

Statistical treatment of random errors; spread, sample and population; sample mean and population mean. Standard deviation and variance of population; Gaussian distribution.

Sample standard deviation, sample variance, standard error of the mean, relative standard deviation, coefficient of variation, pooled standard deviation. Confidence interval.

Student-t statistics, significance testing, null hypothesis, one tailed and two tailed significance tests. Comparing measured results with a known value.

Comparison of two experimental means. Comparison of standard deviation with the F-test. Paired t- test for comparing individual differences. Error in hypothesis testing. Criteria for rejection of an observation - Q test. Problems. Calibration curves: least square method. Finding the least square line. Expression for slope, intercept, standard deviation about regression. Standard deviation of the slope and intercept. Coefficient of determination.

Method validation: Determination limits, calibration sensitivity. limit of quantization and linear dynamic range.

2. Acid – base titrations **6 h**

Basic principles: pH scale, dissociation of acids and bases. Titration curves for mono functional acids and bases, pH calculations, theory of indicators. Titration curves for di,

tri and polybasic acids, polyamines and amino acid systems. Fractions of phosphoric acid species as a function of pH.

Gran's plots application of acid-base titration for environmental, clinical, nutritional and industrial estimations.

3. Redox titrations **8 h**

Nernst equation, standard & formal potentials. Titration curves, end point signals, indicators, criteria for the selection of indicators. Feasibility of redox titration. Titration of multicomponent system. Adjustment of analyte's oxidation state. Application of oxidants such as permanganate, dichromate, Ce (IV), bromate, iodates, and reductants such as ferrous ammonium sulphate and ascorbic acid for environmental, clinical, nutritional and industrial estimations.

Karl-Fischer titrations: Stoichiometry of the reaction, preparation of the reagent, titration method, standardization of the reagent using water-in-methanol, determination of water in samples, interference and their elimination, application to quantitative analysis of some organic compounds such as alcohols, carboxylic acids, acid anhydrides and carbonyl compounds.

References

1. Fundamentals of Analytical Chemistry; Skoog, West. Holler and Crouch 9th edition; Mary Finch. (2014).
2. Analytical Chemistry; Gary D Christian; 6th edition; John Wiley and Sons (2010).
3. Modern Analytical Chemistry David Harvey; McGraw Hill Higher education publishers, (2000).
4. Analytical Chemistry Principles – John H Kennnedy, 2nd edition, Published by Cengage Delmar Learning India Pvt (2011).
5. Principles of Instrumental Analysis, Skoog, Holler and Nieman, 5th edition, Saunders College Publishing, International Limited (1999).
6. Vogel's Text book of quantitative chemical analysis, 6th edition, Pearson Education Limited, (2007).

Course outcomes: After learning this paper students will be able be to

CO1	Remember	Recall concepts, laws and relationships in chemical analysis and data validation and interpretation.
CO2	Understand	Explain the principles of acid-base titrations, redox titrations and instrumental methods of analysis. Describe the experimental methods in chemical analysis.
CO3	Apply	Validate the quality of analytical data using statistical methods. Compute quantities of constituents of samples from experimental data. Construct titration curves of a given data from chemical principles.
CO4	Analyse	Evaluate errors in chemical analysis through statistical treatment of data. Ability to analyse the separation system for multi-component mixtures.
.CO5	Evaluate	Judge the source of the error an experimental result. Compare the quality of two/more statistical treatment. Evaluate sensitivity and selectivity of analytical methods
CO6	Create	Suggest suitable analytical methods for any given system.

Blue print: paper code:

Chapter number	**Title**	**Number of teaching hours (as given in the syllabus)**	**Maximum marks for which questions are to be framed (including extra questions)**
1	Errors in chemical analysis, statistical data treatment and evaluation	16	27
2	Acid-base titrations	6	10
3	Redox titrations	8	13
Total marks excluding extra questions			35
Total marks including extra questions			50

Course title	**SPECTROSCOPY-I**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. Introduction to Spectroscopy **2 h**

Electromagnetic spectrum, relation between energy, wavelength, frequency and wave number; difference between colorimeter and spectrophotometer, types of spectroscopy.

2. UV-Visible Spectroscopy **5 h**

Nature of electronic transitions; the origin of UV band structure; principles of absorption spectroscopy, instrumentation and presentation of spectra. Solvent selection and effect of polar solvent on $\pi - \pi^*$ and $n - \pi^*$ transitions. Terminology: chromophores; auxochromes; bathochromic shift; hypsochromic shift, hyperchromic shift, hypochromic shift. Effect of conjugation on the spectra of alkenes. Electronic spectra of carbonyl compounds.

3. FTIR-Spectroscopy **9 h**

Infrared absorption process. Principle of IR analysis, uses of infrared spectrum. Modes of stretching and bending vibrations. Bond properties and absorption trends. Instrumentation of IR spectrometer: Dispersive and Fourier transform spectrometers. Preparation of samples for IR analysis. Basic analysis of an IR spectrum. Vibration of polyatomic molecules. Survey of functional groups with examples. Hydrocarbons: alkanes, alkenes and alkynes, aromatic hydrocarbons: Detailed discussions on C–H vibrations, C=C vibrations and conjugate effects, Alcohols and phenols, ethers: Detailed discussion on O–H stretching vibration, effect of hydrogen bonding (effect of solvent polarity and concentration). Carbonyl compounds: Normal base values for C=O stretching vibrations for carbonyl compounds. Effect of electron withdrawing groups, inductive, resonance, hydrogen bonding and conjugation. General discussions of IR absorption characteristics of aldehydes, ketones, carboxylic acids, esters, amides, acid anhydrides and chlorides. IR spectra of nitriles, phosphorous compounds, metal-carbonyl complexes. Structure determination of simple molecules.

4. ^{1}H NMR spectroscopy **10 h**

Nuclear spin states; nuclear magnetic moments; absorption of energy; mechanism of absorption (resonance). Population densities of nuclear spin states; The Chemical Shift and shielding; The Nuclear Magnetic Resonance Spectrometer -The Continuous-Wave (CW) Instrument and the Pulsed Fourier Transform (FT) Instrument. NMR instruments with different operating frequencies; Chemical Equivalence; Integrals and Integration; Chemical environment and Chemical shift; Local Diamagnetic Shielding -

Electronegativity Effects ; Hybridization Effects; Acidic and Exchangeable Protons; Hydrogen Bonding. Magnetic Anisotropy; Spin–Spin Splitting (n +1) rule; origin of spin–spin Splitting; Pascal's Triangle. Low and high resolution spectra of ethanol – chemical exchange; NMR spectra of amides. Coupling Constants: The Mechanism of Coupling. Effect of -F on proton NMR. Solving NMR spectra problems.

5. C-13 NMR spectroscopy 4 h

The Carbon-13 Nucleus; Carbon-13 chemical shifts; Proton-Coupled C-13 Spectra—spin–spin splitting of Carbon-13 Signals. Proton-Decoupled C - 13 spectra; Cross-Polarization; Problems with integration in C-13 spectra.

The DEPT experiment: number of protons attached to C - 13 atoms.

Combined spectral problems

References

1. Introduction to Spectroscopy, 5th Edition, Donald L. Pavia, Gary M. Lampman and George S. Kriz and James R. Vyvyan. Cengage Learning (2015).
2. Organic Spectroscopy, W. Kemp, 3rd edition, MacMillan (1991)
3. Spectrometric Identification of Organic Compounds, R.M. Silverstein and W.P. Webster, 8th edition, Wiley & Sons, (2014)
4. Fundamentals of Molecular Spectroscopy C.N. Banwell and E.M. McCash, 4th edition, McGrahill education (2017)
5. Physical methods in Inorganic chemistry, R.S. Drago, Affiliated East-West Press Pvt. Ltd., New Delhi (1965).
6. Principles of Instrumental Analysis, D.A. Skoog, S.J. Holler, T.A. Nilman, 5th Edition, Saunders College Publishing, London, (1998).

Course outcomes: After studying this paper students will be able to

CO1	Knowledge	Recall concepts, laws, relationships in spectroscopic analysis.
CO2	Understand	Explain the concepts, laws, relationships in spectroscopic analysis. Describe the instrumentation of various spectrophotometers. Explain the effect of conjugation, hydrogen bonding, resonance, inductive effect, ring size effect on IR absorption frequency of C=O and C=C functional groups.
CO3	Apply	Calculate coupling constants from proton NMR spectra, and utilize the coupling constants for determining the structure of the compound. Predict the splitting patterns in the proton NMR spectrum of a given compound. Assign peaks in an NMR spectrum to specific protons of a compound.
CO4	Analyze	Interpret spectra of UV-Visible, IR and NMR spectroscopic methods.
CO5	Evaluate	Assess the suitability of the different spectroscopic methods for the structural analysis of a given compound.
CO6	Create	Predict spectra for simple molecules. Deduce the molecular structure of unknown molecule using the combined spectral data.

Blue print: Paper code:

Chapter number	**Title**	**Number of teaching hours**	**Maximum marks for which questions are to be framed (including extra questions)**
1	Introduction to Spectroscopy	2	3
2	UV-Visible Spectroscopy	5	9
3	FTIR-Spectroscopy	9	14
4	^{1}H NMR spectroscopy	10	17
5	C-13 NMR spectroscopy	4	7
Total marks excluding extra questions			35
Total marks including extra questions			50

Course title	**SEPARATION TECHNIQUES**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. Solvent extraction **3 h**

Partition coefficient, equation for batch extraction and multiple extraction, Extraction efficiency- pH effects, membrane assisted solvent extraction (MASE).

2. Theoretical aspects of chromatography **5 h**

Types of chromatography. Theoretical principles - retention time, retention volume, adjusted retention time, relative retention, capacity factor (retention factor), relation between retention time and partition coefficient. Efficiency of separation, resolution - ideal chromatographic peaks (Gaussian peak shape), factors for resolution-diffusion, diffusion coefficient, plate height, plate height as a measure of column efficiency, number of theoretical plates, asymmetric peaks. Factors affecting resolution , band spreading-van Deemter equation, optimum flow rate, A term – multiple paths, longitudinal diffusion, mass transport, extra column contributions to zone broadening.

3. Sample preparation **2h**

Statistics of sampling, choosing a sample size, choosing the number of replicates.

Assignment topic: dissolution and sample preparation of inorganic, organic and biological samples

4. Gas chromatography **6 h**

Separation process in gas chromatography-schematic diagram, open tubular columns and comparison with packed columns. Effect of column inner diameter and length of the column. Choice of liquid stationary phase, molecular sieves as stationary phase, packed columns, retention index, temperature and pressure programming, carrier gas, guard columns and retention gaps, sample injections, split injection and splitless injection. Detectors : thermal conductivity detector, flame ionisation detector, electron capture detector, sample preparation for GC-solid phase microextraction, purge and trap, thermal desorption- Derivatisation in GC-Method development in GC.

5. Liquid chromatography **8 h**

The chromatographic process, effect of small particles, bonded stationary phases, solute column interactions, shape selectivity. The elution process, isocratic and gradient elution, selecting the separation mode, solvents. Maintaining symmetric band shape, dead volume. Injection and detection in HPLC, detector characteristics, signal to noise ratio, detection limits, linearity. Spectrophotometric detectors, refractive index detector, evaporative light scattering detector.
Method development in reverse phase separation, criteria for adequate separation, tailing of peaks, optimization with one solvent, optimization with two different solvents, choosing a stationary phase. Gradient separations. Derivatives for HPLC.

6. Liquid chromatographic methods **4 h**

Normal phase and reverse phase chromatography- retention mechanism and application in isomeric separation
Ion Exchange chromatography- ion exchangers, applications.

Suppressed ion –anion and cation chromatography, Ion chromatography without suppression, detectors, Ion pair chromatography

Size exclusion chromatography-the elution equation, stationary phase, molecular mass determination.

Affinity chromatography- principle-matrix, ligand, spacer arm-properties required for efficient and effective chromatographic matrix, partial structure of agarose. Types of ligands- need of spacer arm. Immobilized metal affinity chromatography.

Chiral separations-Chiral phases (Amylose, crown ethers and cyclodextrins)-Ligand Exchange chromatography, prikle brush type phases.
Supercritical fluid chromatography & hydrophilic interaction chromatography (hilic)

7. Thin layer chromatography **2 h**

Preparation of thin layer plates, sample application, developing chromatogram, visualizing chromatograms, retention factor, high performance thin layer chromatography, forced flow planar chromatography.

References

1. Quantitative Chemical Analysis, Daniel C. Harris, 7th edition., (W. H. Freeman and Company, New York, (2006).
2. Analytical Chemistry Principles – John H Kennnedy, 2nd edition, Published by Cengage Delmar Learning India Pvt (2011).
3. Principles of Instrumental Analysis, Skoog, Holler and Nieman, 5th edition,

Saunders College Publishing, International Limited (1999).

Course outcomes: After learning this paper students will be able to

CO1	Remember	Recall fundamental principles, laws and relationships in solvent extraction and chromatography.
CO2	Understand	Explain fundamental principles, laws and relationships in solvent extraction and chromatography. Describe experimental techniques in solvent extraction and chromatography.
CO3	Apply	Select appropriate method for separation of analytes.
CO4	Analyse	Optimise parameters for separation of analytes.
CO5	Evaluate	Interpret the efficiency of separation techniques for analytes.
CO6	Create	Design suitable method for the effective separation of any given mixture.

Blue print: paper code:

Chapter number	**Title**	**Number of teaching hours (as given in the syllabus)**	**Maximum marks for which questions are to be framed (including extra questions)**
1	Solvent extraction	3	5
2	Theoretical aspects of chromatography	5	8
3	Sample preparation	2	3
4	Gas chromatography	6	10
5	Liquid chromatography	8	14
6	Liquid chromatographic methods	6	10
Total marks excluding extra questions			35
Total marks including extra questions			50

I SEMESTER PRACTICALS

Practical I: Introductory laboratory techniques and practices (Paper code:)

1. Stoichiometric calculations- I
2. Stoichiometric calculations- II
3. Preparation of stock solutions
4. Basic laboratory techniques in sample preparation
5. Statistical methods in analysis

6. Use of laboratory glassware/apparatus
7. Use of equipment
8. Calibration methods
9. Any other suitable experiment

Practical II: Volumetric and gravimetric analysis (paper code:)

1. Estimation of Cu and Ni in an alloy
2. Estimation of total alkalinity (OH^-, carbonate, bicarbonate) of water
3. Estimation of COD of water
4. Estimation of BOD of water
5. Estimation of hardness of water
6. Estimation of chloride by Volhard's method
7. Determination of ascorbic acid in vitamin C tablet
8. Autotitrations (Karl-Fischer titrations) (potentiometry, complexometry, acid-base)
9. Any other suitable experiment

Practical III: Instrumental methods I- Spectroscopy (Paper code:)

1. Estimation of iron by colorimetry
2. Estimation of copper by colorimetry
3. UV-spectral analysis of inorganic and organic compounds
4. Estimation of Cd/Zn/Mg by Atomic Absorption Spectrometry (AAS)
5. Estimation of organic dyes by emission spectrometry
6. Functional group identification of organic compounds by FTIR
7. Combined spectral problems involving UV, IR and NMR data- I
8. Combined spectral problems involving UV, IR and NMR data- II
9. Any other suitable experiment

Practical IV: Instrumental methods II- Chromatography (Paper code:)

1. Identification of amino acids by paper chromatography
2. Identification of carbohydrates by thin layer chromatography
3. Optimization of mobile phase/solvent system for the separation of components of a mixture by TLC
4. Separation of components of a mixture by column chromatography- I
5. Monitoring the progress of an organic reaction by TLC
6. Isolation and extraction of the products obtained in expt. no. 5 using column chromatography
7. Separation of components of a given mixture by Gas Chromatography
8. Separation of components of a given mixture by HPLC
9. Any other suitable experiment

II SEMESTER- THEORY PAPERS

Course title	**CHEMICAL LABORATORY MANAGEMENT AND BEST LAB PRACTICES- II**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. **Managing chemical waste and solvents** **6 h**
   Importance of waste and solvent management, identifying waste and its hazards, standard methods of collecting and storing waste, segregation of waste, treatment and hazard reduction, disposal methods. Incineration.

2. **Assessing chemical hazard and risk in laboratory** **11 h**
   Risk awareness, MSDS, introduction to different toxicity level and their impact, evaluating the toxic risk of laboratory chemicals, assessing flammable, reactive and explosive hazards, assessing physical hazards, assessing bio-hazards, chemical labelling, introducing green chemistry aspects for every laboratory.
3. **Developing strategic/optimal laboratory practices** **4 h**
   Minimizing solvent waste, chemical hygiene in laboratory, maximizing efficiency of lab facilities, reducing pollution
4. **Good Laboratory Practices (GLP) and good manufacturing practices (GMP) 4 h**
   Fundamentals of GLP, regulated and non-regulated GLP, good manufacturing practices (GMP), GLP training, ICH guidelines
5. **Rules for conduct of studies** **5 h**
   General aspects, study plan of protocol, content of the protocol, identification, approval of protocol, protocol amendment, Standard operating procedures (SOP), system overview, intellectual property rights (IPR).

References

1. Chemistry Laboratory Safety and Security, A Guide Prudent Chemical Management; Lisa Moran and Tina Masciangioli; National Academic press. (2010).
2. Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards; Dr. Ralph J.Cicerone and Dr. Charles M. Vest; ISBN 978-0-309-13864-2; National Academic press.
3. Handbook- Good laboratory practices; Dr R. Ridley, Published by WHO and TDR (2001).

Course outcomes: After studying this paper students will be able to

CO1	Knowledge	Recall good laboratory practices, toxicity and hazard of chemicals, management of chemical waste, optimal laboratory practices, rules for conduct of studies
CO2	Understand	Explain methods involved in waste management, toxicity of laboratory chemicals, hazards of chemicals, green chemistry aspects, fundamentals of good laboratory/manufacturing practices , standard operating procedure (SOP), Intellectual Property Rights (IPR)
CO3	Apply	Develop content of the protocol and standard operating procedures, Develop methods to minimize solvent waste, reduce pollution Develop methods to maximize chemical hygiene and efficiency of lab facilities.
CO4	Analyze	Compare the toxicity/hazard levels of various chemicals in the laboratory
CO5	Evaluate	Assess the laboratory practices and suggest ways to minimize waste and maximize efficiency
CO6	Create	Apply green chemistry principles and design a chemical laboratory with minimum hazard/pollution

Blue print: paper code:

Chapter number	**Title**	**Number of teaching hours**	**Maximum marks for which questions are to be framed**
1	Managing chemical waste and solvents	6	9
2	Assessing chemical hazard and risk in laboratory	11	19
3	Developing strategic/optimal laboratory practices	4	7
4	Good laboratory practices and good management practices	4	7
5	Rules for conduct of studies	5	8
Total marks excluding extra questions			35
Total marks including extra questions			50

Course title	**PRINCIPLES OF CHEMICAL ANALYSIS- II**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. Complexometric titrations **7 h**

Complexometric titrations with particular reference to EDTA titrations, suitability of polydentate ligands as titrants, expressions for the different forms of EDTA in solution as a function of pH, conditional stability constants, derivation of titration curve, effect of pH and second complexing agent on the conditional stability constant and titration curve. Selectivity by pH control, masking and demasking, metal ion indicators, types of EDTA titrations, titrations involving monodentate ligands. Application of EDTA titration for environmental, clinical, nutritional and industrial estimations.

2. Precipitation titrations **4 h**

Solubility product. Theoretical principles: Titration curves, end point signals, Mohr, Volhard and adsorption indicators. Applications of argentometric titrations in estimation of F^-, K^+, CO_3^{2-}, $C_2O_4^{2-}$, acetylenes and mixture of halides.

3. Non-aqueous titrations **4 h**

Acid–base titrations in non-aqueous solvents - acidic and basic titrants, methods of titration. Titrations in glacial acetic acid and ethylene diamine, applications of non-aqueous titrations.

4. Gravimetric analysis **4 h**

Principles of gravimetric analysis, types of gravimetric analysis, different steps involved in gravimetric estimation. Formation and treatment of precipitates, co-precipitation, post precipitation, precipitation from homogeneous solution, important precipitating agents and their significance in inorganic analysis.

5. Kinetic methods of analysis **4 h**

Equilibrium and kinetic methods. Rate laws, pseudo first order kinetics, types of kinetic methods, fixed time methods. Applications of catalytic and non-catalytic kinetic methods.

6. Absorption and Emission techniques **5 h**

Quantitative aspects of spectrochemical Measurements.

Molecular luminescence- explanation for fluorescence and phosphorescence using Jablonski diagram. Quantitative aspects of fluorescence. Interpretation- Internal conversion, vibrational relaxation and intersystem crossing. Fluorescence and structure with examples, effects of temperature, dissolved oxygen and solvent. Instrumentation.

7. Thermal methods of analysis **2 h**

Thermogravimetric analysis - Differential Thermal Analysis; Differential Scanning calorimetry- thermometric titrations and applications.

References

1. Fundamentals of Analytical Chemistry; Skoog, West. Holler and Crouch 9th edition; Mary Finch. (2014).
2. Analytical Chemistry; Gary D Christian; 6th edition; John Wiley and Sons (2010).
3. Modern Analytical Chemistry David Harvey; McGraw Hill Higher education publishers, (2000).
4. Analytical Chemistry Principles – John H Kennnedy, 2nd edition, Published by Cengage Delmar Learning India Pvt (2011).
5. Principles of Instrumental Analysis, Skoog, Holler and Nieman, 5th edition, Saunders College Publishing, International Limited (1999).
6. Vogel's Text book of quantitative chemical analysis, 6th edition, Pearson Education Limited, (2007).

Course outcomes: After learning this paper students will be able to

CO1	Remember	Recall concepts, laws and relationships in chemical analysis
CO2	Understand	Explain the principles of different wet-chemical and instrumental methods of analysis. Describe the experimental methods in chemical analysis.
CO3	Apply	Distinguish between equilibrium and kinetic methods. Compute quantities of constituents of samples from experimental data. Analyze thermal behavior of different organic and inorganic materials using TGA, DTA
CO4	Analyse	Ability to analyse the separation system for multi-component mixtures.
CO5	Evaluate	Evaluate sensitivity and selectivity of analytical methods
CO6	Create	Suggest suitable analytical methods for any given system.

Blue print: paper code:

Chapter number	Title	Number of teaching hours	Maximum marks for which questions are to be framed
1	Complexometric titrations	7	11
2	Precipitation titrations	4	7
3	Non aqueous titrations	4	7
4	Gravimetric analysis	4	7
5	Kinetic methods of analysis	4	7
6	Absorption techniques	5	8
7	Thermal methods of analysis	2	3
Total marks excluding extra questions			35
Total marks including extra questions			50

Course title	**SPECTROSCOPY- II**
Paper code	
Number of teaching hrs per week	2
Total number of teaching hrs per semester	30
Number of credits	2

1. Mass spectrometry **13 h**

Principle of mass spectrometry, mass spectrometer, resolution of mass spectrum, molecular ion peak, base peak, fragment ion peaks, meta stable ion peak, isotope peaks, Nitrogen rule - definition and their significance. Determination of molecular weight and molecular formula. Carbocation: stability, types of fragmentation patterns: single bond, multiple bonds, McLafforty rearrangement, retro Diels- Alder. General discussions on the fragmentation patterns of alkanes, alkenes, aromatic hydrocarbons, alcohols, phenols, ethers, aldehydes, ketones, esters, carboxylic acids, amines. Different ionization and analysis methods: EI, CI, FAB, MALDI, etc. soft ionization and hard ionization techniques-ESI, APCI, APPI- Coulombic explosion, Rayleigh limit. Analysers and detectors. Structure determination of molecules. GC-MS and LC-MS.

2. Atomic Absorption and Emission Spectroscopy **7 h**

Atomic absorption methods- principle and Instrumentation (single and double beam) Light sources of AAS, atomization (flame and electrothermal), Interferences, detection limits, Atomic emission method (AES) Plasma – DCP and ICP-MS and OES techniques.

3. X-Ray Diffraction **10 h**

X-rays, Bragg's equation and Bragg's method, Miller indices, unit cell parameters and (Mentioning of crystal systems whenever required). X-ray structural analysis of solid substances: powder diffraction pattern of primitive, face- centered and body centered cubic lattices, indexing of reflections, identification of space groups from systematic absences (space group extinctions). The concept of reciprocal lattice and construction of Ewald's sphere, derivation of Bragg's law from reciprocal lattice, structure factor(s) and its relation to intensity, intensities from atomic positions for BCC and FCC lattices. Phase problem-heavy atom (Patterson's) method and introduction to the principle of direct methods of phase determination. Electron density function and Fourier synthesis, electron density map(s).

References

1. Fundamentals of Analytical Chemistry; Skoog, West. Holler and Crouch 9th edition; Mary Finch. (2014).
2. Organic Mass Spectroscopy, K.R. Dass and E.P. James, IBH New Delhi, (1976).
3. Mass Spectrometry of Organic Compounds, H. Budzkiewicz, Djerassi C. and D.H Williams, Holden-Day, New York, (1975).
4. Analytical Chemistry; Gary D Christian; 6th edition; John Wiley and Sons (2010).
5. Modern Analytical Chemistry David Harvey; McGraw Hill Higher education publishers, (2000).
6. Analytical Chemistry Principles – John H Kennnedy, 2nd edition, Published by Cengage Delmar Learning India Pvt (2011).
7. Principles of Instrumental Analysis, Skoog, Holler and Nieman, 5th edition, Saunders College Publishing, International Limited (1999).
8. Vogel's Text book of quantitative chemical analysis, 6th edition, Pearson Education Limited, (2007).

Course Outcomes: After studying this paper students will be able to

CO1	Knowledge	Recall concepts, laws, relationships in spectroscopic and X-ray diffraction analysis.
CO2	Understand	Explain the concepts, laws, relationships in spectroscopic and X-ray diffraction methods of analysis. Describe the instrumentation of mass, atomic absorption/ emission spectrometers. Correlate carbocation stability with fragmentation pattern in mass spectra. Describe the quantitative estimation of elements by AAS/AES
CO3	Apply	Fragmentation pattern of molecules with C=O and C=N using McLafforty rearrangement. Predict the crystal structure by XRD Compute *d*-spacing from X-ray diffractogram
CO4	Analyze	Interpret Mass spectra and X-ray diffractogram
CO5	Evaluate	Assess the suitability of the different spectroscopic methods for the determine composition and crystal structure of a given compound.
CO6	Create	Predict molecular formula from mass spectral data

Blue print: paper code:

Chapter number	Title	Number of teaching hours	Maximum marks for which questions are to be framed
1	Mass spectrometry	13	22
2	Atomic absorption and Emission spectroscopy	7	11
3	X-ray diffraction	10	17
Total marks excluding extra questions			35
Total marks including extra questions			50

PRACTICALS

Practical V- Application of software in Chemistry

Chemdraw, chemsketch, chemspider, origin, using experimental data and plotting line of best fit in origin, error analysis using excel, search engines- google scholar, google patent, espacenet, uspto, scopus, scifinder ; inflibnet, jstor

Practical VI- Instrumental methods III- Assorted techniques

1. Estimation of transition metal ions by potentiometry
2. Conductometric estimation of urea by hydrolysis
3. Non-aqueous titrations- autotitration (Estimation of nicotine in tobacco leaves)
4. Determination of optimum pH for enzymatic activity
5. Determination of melting point
6. Determination of optical rotation by polarimetry
7. Determination of density, refractive index and viscosity of liquids
8. Determination of water of hydration in a compound by thermogravimetry
9. Determination of melting point of samples by Differential Scanning Calorimetry

Practical VII- Instrumental methods IV - Spectroscopy- Quantitative estimations and validation methods(9 sessions)

Practical VIII- Instrumental methods V- Chromatography- Quantitative estimations and validation methods (9 sessions)