

Vidyasagar University

Curriculum for B.Sc. (Honours) in Chemistry [Choice Based Credit System]

Semester-V

Course	Course Code	Name of the Subjects	Course Type/ Nature	Teaching Scheme in hour per week			Credit	Marks
				L	T	P		
CC- 11		C11T: Inorganic Chemistry - IV	Core Course-11	4	0	0	6	75
		- Lab		0	0	4		
CC- 12		C12T: Organic Chemistry - V	Core Course-12	4	0	0	6	75
		- Lab		0	0	4		
DSE-1		DSE1T: Advanced Physical Chemistry	Discipline Specific Electives -1	4	0	0	6	75
		- Lab		0	0	4		
DSE-2		DSE2T: Analytical Methods in Chemistry Or Instrumental Methods of Chemical Analysis	Discipline Specific Electives -2	4	0	0	6	75
		- Lab		0	0	4		
Semester Total							24	300

L= Lecture, T= Tutorial, P = Practical, CC - Core Course, TBD - To be decided, DSE: Discipline Specific Elective.

Semester-V

List of Core Course (CC)

CC-11: Inorganic Chemistry - IV

CC-12: Organic Chemistry - V

Discipline Specific Electives (DSE)

DSE-1: Advanced Physical Chemistry

DSE-2: Analytical Methods in Chemistry

Or

DSE-2: Instrumental Methods of Chemical Analysis

SEMESTER –V
Core Courses (CC)

CC-11: Inorganic Chemistry - IV

Credits 06

C11T: Inorganic Chemistry - IV

Credits 04

Course Contents:

Coordination Chemistry-II

VB description and its limitations. Elementary Crystal Field Theory: splitting of d^n configurations in octahedral, square planar and tetrahedral fields, crystal field stabilization energy (CFSE) in weak and strong fields; pairing energy. Spectrochemical series. Jahn- Teller distortion. Octahedral site stabilization energy (OSSE). Metal-ligand bonding (MO concept, elementary idea), sigma- and pi-bonding in octahedral complexes (qualitative pictorial approach) and their effects on the oxidation states of transitional metals (examples). Magnetism and Colour: Orbital and spin magnetic moments, spin only moments of d^n ions and their correlation with effective magnetic moments, including orbital contribution; quenching of magnetic moment: super exchange and antiferromagnetic interactions (elementary idea with examples only); d-d transitions; L-S coupling; qualitative Orgel diagrams for $3d^1$ to $3d^9$ ions. Racah parameter. Selection rules for electronic spectral transitions; spectrochemical series of ligands; charge transfer spectra (elementary idea).

Chemistry of d- and f- block elements

Transition Elements:

General comparison of 3d, 4d and 5d elements in term of electronic configuration, oxidation states, redox properties, coordination chemistry.

Lanthanoids and Actinoids:

General Comparison on Electronic configuration, oxidation states, colour, spectral and magnetic properties; lanthanide contraction, separation of lanthanides (ion-exchange method only).

Suggested Readings:

1. Huheey, J. E.; Keiter, E.A. & Keiter, R.L. *Inorganic Chemistry, Principles of Structure and Reactivity 4th Ed.*, Harper Collins 1993, Pearson, 2006.
2. Greenwood, N.N. & Earnshaw A. *Chemistry of the Elements*, Butterworth-Heinemann. 1997.
3. Cotton, F.A., Wilkinson, G., Murrillo, C. A., Bochmann, M., *Advanced Inorganic Chemistry 6th Ed.* 1999., Wiley.

4. Atkin, P. *Shriver & Atkins' Inorganic Chemistry* 5th Ed. Oxford University Press (2010).
5. Purecell, K.F. and Kotz, J.C., *An Introduction to Inorganic Chemistry*, Saunders: Philadelphia, 1980.
6. Sinha, S. P., Ed., *Lanthanide and Actinide Research* (Journal, Vol. 1, 1986).
7. Wulfsberg, G., *Principles of Descriptive Inorganic Chemistry*, Brooks/Cole: Monterey, CA, 1987.

C11P : LAB

Credits 02

Practicals :

Chromatography of metal ions

Principles involved in chromatographic separations. Paper chromatographic separation of following metal ions:

1. Ni (II) and Co (II)
2. Fe (III) and Al (III)

Gravimetry

1. Estimation of Ni(II) using Dimethylglyoxime (DMG).
2. Estimation of copper as CuSCN.
3. Estimation of Al(III) by precipitating with oxine and weighing as Al(oxine)₃ (aluminium oxinate).
4. Estimation of chloride.

Spectrophotometry

1. Measurement of 10Dq by spectrophotometric method.
2. Determination of λ_{\max} of [Mn(acac)₃] and [Fe(acac)₃] complexes.

Suggested Readings:

1. Mendham, J., *A. I. Vogel's Quantitative Chemical Analysis* 6th Ed., Pearson, 2009.

CC-12: Organic Chemistry - V

Credits 06

C12T: Organic Chemistry - V

Credits 04

Course Contents:

Carbocycles and Heterocycles

Polynuclear hydrocarbons and their derivatives: synthetic methods include Haworth, Bardhan-Sengupta, Bogert-Cook and other useful syntheses (with mechanistic details); fixation of double bonds and Fries rule; reactions (with mechanism) of naphthalene, anthracene, phenanthrene and their derivatives.

Heterocyclic compounds: 5- and 6-membered rings with one heteroatom; reactivity, orientation and important reactions (with mechanism) of furan, pyrrole, thiophene and pyridine; synthesis (including retrosynthetic approach and mechanistic details): pyrrole: Knorr synthesis, Paal-Knorr synthesis, Hantzsch; furan: Paal-Knorr synthesis, Feist-Benary synthesis and its variation; thiophenes: Paal-Knorr synthesis, Hinsberg synthesis; pyridine: Hantzsch synthesis; benzo-fused 5- and 6-membered rings with one heteroatom: reactivity, orientation and important reactions (with mechanistic details) of indole, quinoline and isoquinoline; synthesis (including retrosynthetic approach and mechanistic details): indole: Fischer, Madelung and Reissert; quinoline: Skraup, Doebner- Miller, Friedlander; isoquinoline: Bischler-Napieralski synthesis.

Cyclic Stereochemistry

Alicyclic compounds: concept of I-strain; conformational analysis: cyclohexane, mono and disubstituted cyclohexane; symmetry properties and optical activity; topomerisation; ring-size and ease of cyclisation; conformation & reactivity in cyclohexane system: consideration of steric and stereoelectronic requirements; elimination (E2, E1), nucleophilic substitution (S_N1, S_N2, S_Ni, NGP), merged substitution-elimination; rearrangements; oxidation of cyclohexanol, esterification, saponification, lactonisation, epoxidation, pyrolytic *syn* elimination and fragmentation reactions.

Pericyclic reactions

Mechanism, stereochemistry, regioselectivity in case of

Electrocyclic reactions: FMO approach involving 4 π - and 6 π -electrons (thermal and photochemical) and corresponding cycloreversion reactions.

Cycloaddition reactions: FMO approach, Diels-Alder reaction, photochemical [2+2] cycloadditions.

Sigmatropic reactions: FMO approach, sigmatropic shifts and their order; [1,3]- and [1,5]-H shifts and [3,3]-shifts with reference to Claisen and Cope rearrangements.

Carbohydrates

Monosaccharides: Aldoses up to 6 carbons; structure of D-glucose & D-fructose (configuration & conformation); ring structure of monosaccharides (furanose and pyranose forms): Haworth representations and non-planar conformations; anomeric effect (including stereoelectronic explanation); mutarotation; epimerization; reactions (mechanisms in relevant cases): Fischer glycosidation, osazone formation, bromine-water oxidation, HNO₃ oxidation, selective oxidation of terminal –CH₂OH of aldoses, reduction to alditols, Lobry de Bruyn-van Ekenstein rearrangement; stepping-up (Kiliani-Fischer method) and stepping-down (Ruff's & Wohl's methods) of aldoses;

end-group-interchange of aldoses; acetonide (isopropylidene) and benzylidene protections; ring-size determination; Fischer's proof of configuration of (+)-glucose.

Disaccharides: Glycosidic linkages, concept of glycosidic bond formation by glycosyl donor-acceptor; structure of sucrose, inversion of cane sugar.

Polysaccharides: starch (structure and its use as an indicator in titrimetric analysis).

Bio-molecules

Amino acids: synthesis with mechanistic details: Strecker, Gabriel, acetamido malonic ester, azlactone, Bücherer hydantoin synthesis, synthesis involving diketopiperazine; isoelectric point, zwitterions; electrophoresis, reaction (with mechanism): ninhydrin reaction, Dakin-West reaction; resolution of racemic amino acids.

Peptides: peptide linkage and its geometry; syntheses (with mechanistic details) of peptides using *N*-protection & *C*-protection, solid-phase (Merrifield) synthesis; peptide sequence: *C*-terminal and *N*-terminal unit determination (Edman, Sanger & 'dansyl' methods); partial hydrolysis; specific cleavage of peptides: use of CNBr.

Nucleic acids: pyrimidine and purine bases (only structure & nomenclature); nucleosides and nucleotides corresponding to DNA and RNA; mechanism for acid catalysed hydrolysis of nucleosides (both pyrimidine and purine types); comparison of alkaline hydrolysis of DNA and RNA; elementary idea of double helical structure of DNA (Watson-Crick model); complimentary base-pairing in DNA.

Suggested Readings:

1. Clayden, J., Greeves, N., Warren, S. *Organic Chemistry*, Second edition, Oxford University Press 2012.
2. Eliel, E. L. & Wilen, S. H. *Stereochemistry of Organic Compounds*, Wiley: London.
3. Nasipuri, D. *Stereochemistry of Organic Compounds*, Wiley Eastern Limited.
4. Fleming, I. *Molecular Orbitals and Organic Chemical reactions*, Reference/Student Edition, Wiley, 2009.
5. Fleming, I. *Pericyclic Reactions*, Oxford Chemistry Primer, Oxford University Press.
6. Gilchrist, T. L. & Storr, R. C. *Organic Reactions and Orbital symmetry*, Cambridge University Press.
7. Finar, I. L. *Organic Chemistry (Volume 1)*, Dorling Kindersley (India) Pvt. Ltd.(Pearson Education).
8. Finar, I. L. *Organic Chemistry (Volume 2: Stereochemistry and the Chemistry of Natural Products)*, Dorling Kindersley (India) Pvt. Ltd. (Pearson Education).
9. Morrison, R. T. & Boyd, R. N. *Organic Chemistry*, Dorling Kindersley (India) Pvt. Ltd. (Pearson Education).
10. Loudon, G. M. *Organic Chemistry*, Fourth edition, Oxford University Press.

11. James, J., Peach, J. M. *Stereochemistry at a Glance*, Blackwell Publishing, 2003.
12. Robinson, M. J. T., *Stereochemistry*, Oxford Chemistry Primer, Oxford University Press, 2005.
13. Davis, B. G., Fairbanks, A. J., *Carbohydrate Chemistry*, Oxford Chemistry Primer, Oxford University Press.
14. Joule, J. A. Mills, K. *Heterocyclic Chemistry*, Blackwell Science.
15. Acheson, R.M. *Introduction to the Chemistry of Heterocyclic compounds*, John Wiley & Sons (1976).
16. Gilchrist, T. L. *Heterocyclic Chemistry*, 3rd edition, Pearson.
17. Davies, D. T., *Heterocyclic Chemistry*, Oxford Chemistry Primer, Oxford University Press.
18. Organic Chemistry, Paula Bruice

C12P : LAB

Credits 06

Practicals :

A. Chromatographic Separations

1. TLC separation of a mixture containing 2/3 amino acids
2. TLC separation of a mixture of dyes (fluorescein and methylene blue)
3. Column chromatographic separation of leaf pigments from spinach leaves
4. Column chromatographic separation of mixture of dyes
5. Paper chromatographic separation of a mixture containing 2/3 amino acids
6. Paper chromatographic separation of a mixture containing 2/3 sugars

B. Spectroscopic Analysis of Organic Compounds

1. Assignment of labelled peaks in the ^1H NMR spectra of the known organic compounds explaining the relative δ -values and splitting pattern.
2. Assignment of labelled peaks in the IR spectrum of the same compound explaining the relative frequencies of the absorptions (C-H, O-H, N-H, C-O, C-N, C-X, C=C, C=O, N=O, C \equiv C, C \equiv N stretching frequencies; characteristic bending vibrations are included).
3. The students must record full spectral analysis of **at least 15 (fifteen)** compounds from the following list:
 - (i) 4'-Bromoacetanilide (ii) 2-Bromo-4'-methylacetophenone (iii) Vanillin (iv) 2'-Methoxyacetophenone (v) 4-Aminobenzoic acid (vi) Salicylamide (vii) 2'-Hydroxyacetophenone (viii) 1,3-Dinitrobenzene (ix) *trans*-Cinnamic acid (x) *trans*-4-Nitrocinnamaldehyde (xi) Diethyl fumarate (xii) 4-Nitrobenzaldehyde (xiii) 4'-Methylacetanilide (xiv) Mesityl oxide (xv) 2-Hydroxybenzaldehyde (xvi) 4-Nitroaniline (xvii) 2-Hydroxy-3-nitrobenzaldehyde (xviii) 2,3-Dimethylbenzoinitrile (xix) Pent-1-yn-3-ol (xx) 3-Nitrobenzaldehyde (xxi) 3-Ethoxy-4-hydroxybenzaldehyde (xxii) 2-Methoxybenzaldehyde (xxiii) Methyl 4-hydroxybenzoate (xxiv) Methyl 3-hydroxybenzoate (xxv) 3-Aminobenzoic acid (xxvi) Ethyl 3-aminobenzoate (xxvii) Ethyl 4-aminobenzoate (xxviii) 3-nitroanisole (xxix) 5-Methyl-2-nitroanisole (xxx) 3'-Methylacetanilide

Suggested Readings:

1. *University Hand Book of Undergraduate Chemistry Experiments*, edited by Mukherjee, G. N. University of Calcutta, 2003.
2. *Practical Workbook Chemistry (Honours), UGBS, Chemistry*, University of Calcutta, 2015
3. Furniss, B.S.; Hannaford, A.J.; Smith, P.W.G.; Tatchell, A.R. *Practical Organic Chemistry, 5th Ed.*, Pearson (2012).
4. Mann, F.G. & Saunders, B.C. *Practical Organic Chemistry*, Pearson Education.

Discipline Specific Electives (DSE)

DSE -1: Advanced Physical Chemistry

Credits 06

DSE1T: Advanced Physical Chemistry

Credits 04

Course Contents:

a) Crystal Structure

Bravais Lattice and Laws of Crystallography: Types of solid, Bragg's law of diffraction; Laws of crystallography (Haüy's law and Steno's law); Permissible symmetry axes in crystals; Lattice, space lattice, unit cell, crystal planes, Bravais lattice. Packing of uniform hard sphere, close packed arrangements (fcc and hcp); Tetrahedral and octahedral voids. Void space in p-type, F-type and I-type cubic systems

Crystal planes: Distance between consecutive planes [cubic, tetragonal and orthorhombic lattices]; Indexing of planes, Miller indices; calculation of d_{hkl} ; Relation between molar mass and unit cell dimension for cubic system; Bragg's law (derivation)

Determination of crystal structure: Powder method; Structure of NaCl and KCl crystals

b) Statistical Thermodynamics

Configuration: Macrostates, microstates and configuration; calculation with harmonic oscillator; variation of W with E ; equilibrium configuration

Boltzmann distribution: Thermodynamic probability, entropy and probability, Boltzmann distribution formula (with derivation); Applications to barometric distribution; Partition function, concept of ensemble - canonical ensemble and grand canonical ensembles

Partition function: molecular partition function and thermodynamic properties, Maxwell's speed distribution; Gibbs' paradox

c) Special selected topics

Specific heat of solid: Coefficient of thermal expansion, thermal compressibility of solids; Dulong –Petit’s law; Perfect Crystal model, Einstein’s theory – derivation from partition function, limitations; Debye’s T^3 law – analysis at the two extremes

3rd law: Absolute entropy, Plank’s law, Calculation of entropy, Nernst heat theorem

Adiabatic demagnetization: Approach to zero Kelvin, adiabatic cooling, demagnetization, adiabatic demagnetization – involved curves

Polymers: Classification of polymers, nomenclature, Molecular forces and chemical bonding in polymers, Texture of Polymers; Criteria for synthetic polymer formation; Relationships between functionality, extent of reaction and degree of polymerization; Mechanism and kinetics of step growth and copolymerization; Conducting polymers

Suggested Readings:

1. Castellan, G. W. *Physical Chemistry*, Narosa
2. Levine, I. N. *Physical Chemistry*, Tata McGraw-Hill
3. Moore, W. J. *Physical Chemistry*, Orient Longman
4. Atkins, P. W. & Paula, J. de *Atkins’ Physical Chemistry*, Oxford University Press
5. McQuarrie, D. A. & Simons, J. D. *Physical Chemistry: A Molecular Approach*, Viva Press
6. Engel, T. & Reid, P. *Physical Chemistry*, Pearson
7. Nash, L. K. *Elements of Statistical Thermodynamics*, Dover
8. Rastogi, R. P. & Misra, R.R. *An Introduction to Chemical Thermodynamics*, Vikas
9. Zemansky, M. W. & Dittman, R.H. *Heat and Thermodynamics*, Tata-McGraw-Hill
10. Billmeyer, F. W. *Textbook of Polymer Science*, John Wiley & Sons, Inc.
11. Seymour, R. B. & Carraher, C. E. *Polymer Chemistry: An Introduction*, Marcel Dekker, Inc.
12. Odian, G. *Principles of Polymerization*, Wiley
13. Billmeyer, F. W. *Textbook of Polymer Science*, Wiley Interscience, 1971.

DSE1P: Advanced Physical Chemistry (Lab)

Credits 02

Practicals :

Computer programs based on numerical methods for

Programming 1: Roots of equations: (e.g. volume of van der Waals gas and comparison with ideal gas, pH of a weak acid)

Programming 2: Numerical differentiation (e.g., change in pressure for small change in volume of a van der Waals gas, potentiometric titrations)

Programming 3: Numerical integration (e.g. entropy/ enthalpy change from heat capacity data), probability distributions (gas kinetic theory) and mean values

Programming 4: Matrix operations (Application of Gauss-Siedel method in colourimetry)

Programming 5: Simple exercises using molecular visualization software

Suggested Readings:

1. Mc Quarrie, D. A. *Mathematics for Physical Chemistry* University Science Books (2008)
2. Mortimer, R. *Mathematics for Physical Chemistry*. 3rd Ed. Elsevier (2005)
3. Yates, P. *Chemical Calculations*. 2nd Ed. CRC Press (2007)
4. Harris, D. C. *Quantitative Chemical Analysis*. 6th Ed., Freeman (2007) Chapters 3-5
5. Noggle, J. H. *Physical Chemistry on a Microcomputer*. Little Brown & Co. (1985)

DSE-2: Analytical Methods in Chemistry

Credits 06

DSE2T: Analytical Methods in Chemistry

Credits 04

Course Contents:

Qualitative and quantitative aspects of analysis:

Sampling, evaluation of analytical data, errors, accuracy and precision, methods of their expression, normal law of distribution if indeterminate errors, statistical test of data; F, Q and t test, rejection of data, and confidence intervals.

Optical methods of analysis:

Origin of spectra, interaction of radiation with matter, fundamental laws of spectroscopy and selection rules, validity of Beer-Lambert's law.

UV-Visible Spectrometry: Basic principles of instrumentation (choice of source, monochromator and detector) for single and double beam instrument;

Basic principles of quantitative analysis: estimation of metal ions from aqueous solution, geometrical isomers, keto-enol tautomers. Determination of composition of metal complexes using Job's method of continuous variation and mole ratio method.

Infrared Spectrometry: Basic principles of instrumentation (choice of source, monochromator & detector) for single and double beam instrument; sampling techniques.

Structural illustration through interpretation of data, Effect and importance of isotope substitution.

Flame Atomic Absorption and Emission Spectrometry: Basic principles of instrumentation (choice of source, monochromator, detector, choice of flame and Burner designs. Techniques of atomization and sample introduction; Method of background correction, sources of chemical interferences and their method of removal. Techniques for the quantitative estimation of trace level of metal ions from water samples.

Thermal methods of analysis:

Theory of thermogravimetry (TG), basic principle of instrumentation.

Techniques for quantitative estimation of Ca and Mg from their mixture.

Electroanalytical methods:

Classification of electroanalytical methods, basic principle of pH metric, potentiometric and conductometric titrations. Techniques used for the determination of equivalence points. Techniques used for the determination of pKa values.

Separation techniques:

Solvent extraction: Classification, principle and efficiency of the technique.

Mechanism of extraction: extraction by solvation and chelation.

Technique of extraction: batch, continuous and counter current extractions.

Qualitative and quantitative aspects of solvent extraction: extraction of metal ions from aqueous solution, extraction of organic species from the aqueous and nonaqueous media.

Chromatography: Classification, principle and efficiency of the technique.

Mechanism of separation: adsorption, partition & ion exchange.

Development of chromatograms: frontal, elution and displacement methods.

Qualitative and quantitative aspects of chromatographic methods of analysis: IC, GLC, GPC, TLC and HPLC.

Stereoisomeric separation and analysis: Measurement of optical rotation, calculation of Enantiomeric excess (ee)/ diastereomeric excess (de) ratios and determination of enantiomeric composition using NMR, Chiral solvents and chiral shift reagents. Chiral chromatographic techniques using chiral columns (GC and HPLC).

Role of computers in instrumental methods of analysis.

Suggested Readings:

1. Mendham, J., A. I. Vogel's *Quantitative Chemical Analysis 6th Ed.*, Pearson, 2009.
2. Willard, H.H. *et al.*: *Instrumental Methods of Analysis*, 7th Ed. Wardsworth Publishing Company, Belmont, California, USA, 1988.
3. Christian, G.D. *Analytical Chemistry*, 6th Ed. John Wiley & Sons, New York, 2004.
4. Harris, D.C.: *Exploring Chemical Analysis*, 9th Ed. New York, W.H. Freeman, 2016.
5. Khopkar, S.M. *Basic Concepts of Analytical Chemistry*. New Age International Publisher, 2009.
6. Skoog, D.A. Holler F.J. & Nieman, T.A. *Principles of Instrumental Analysis*, Cengage Learning India Ed.

7. Mikes, O. *Laboratory Hand Book of Chromatographic & Allied Methods*, Elles Harwood Series on Analytical Chemistry, John Wiley & Sons, 1979.
8. Ditts, R.V. *Analytical Chemistry; Methods of separation*, van Nostrand, 1974.

DSE2P: Analytical Methods in Chemistry (lab)

Credits 02

Practical :

I. Separation Techniques

Chromatography:

- (a) Separation of mixtures

Separation and identification of the monosaccharides present in the given mixture (glucose & fructose) by paper chromatography. Reporting the R_f values.

- (b) Separate a mixture of Sudan yellow and Sudan Red by TLC technique and identify them on the basis of their R_f values.

- (c) Chromatographic separation of the active ingredients of plants, flowers and juices by TLC

II. Solvent Extractions:

To separate a mixture of Ni^{2+} & Fe^{2+} by complexation with DMG and extracting the Ni^{2+} -DMG complex in chloroform, and determine its concentration by spectrophotometry.

Analysis of soil:

- (i) Determination of pH of soil.
- (ii) Estimation of calcium, magnesium, phosphate

Ion exchange:

Determination of exchange capacity of cation exchange resins and anion exchange resins.

III. Spectrophotometry

1. Determination of pKa values of indicator using spectrophotometry.
2. Determination of chemical oxygen demand (COD).
3. Determination of Biological oxygen demand (BOD).

Suggested Readings:

1. Mendham, J., A. I. *Vogel's Quantitative Chemical Analysis 6th Ed.*, Pearson, 2009.
2. Willard, H.H. *et al.: Instrumental Methods of Analysis*, 7th Ed. Wardsworth Publishing Company, Belmont, California, USA, 1988.
3. Christian, G.D. *Analytical Chemistry*, 6th Ed. John Wiley & Sons, New York, 2004.
4. Harris, D.C. *Exploring Chemical Analysis*, 9th Ed. New York, W.H. Freeman, 2016.

5. Khopkar, S.M. *Basic Concepts of Analytical Chemistry*. New Age International Publisher, 2009.
6. Skoog, D.A. Holler F.J. and Nieman, T.A. *Principles of Instrumental Analysis*, Cengage Learning India Edition.
7. Mikes, O. & Chalmes, R.A. *Laboratory Handbook of Chromatographic & Allied Methods*, Elles Harwood Ltd. London.
8. Ditts, R.V. *Analytical Chemistry: Methods of separation*. Van Nostrand, New York, 1974.

Or

DSE-2: Instrumental Methods of Chemical Analysis

Credits 06

DSE2T: Instrumental Methods of Chemical Analysis

Credits 04

Course Contents:

Introduction to spectroscopic methods of analysis:

Recap of the spectroscopic methods covered in detail in the core chemistry syllabus: Treatment of analytical data, including error analysis. Classification of analytical methods and the types of instrumental methods. Consideration of electromagnetic radiation.

Molecular spectroscopy:

Infrared spectroscopy:

Interactions with molecules: absorption and scattering. Means of excitation (light sources), separation of spectrum (wavelength dispersion, time resolution), detection of the signal (heat, differential detection), interpretation of spectrum (qualitative, mixtures, resolution), advantages of Fourier Transform (FTIR). Samples and results expected. Applications: Issues of quality assurance and quality control, Special problems for portable instrumentation and rapid detection.

UV-Visible/ Near IR – emission, absorption, fluorescence and photoacoustic. Excitation sources (lasers, time resolution), wavelength dispersion (gratings, prisms, interference filters, laser, placement of sample relative to dispersion, resolution), Detection of signal (photocells, photomultipliers, diode arrays, sensitivity and S/N), Single and Double Beam instruments, Interpretation (quantification, mixtures, absorption vs. fluorescence and the use of time, photoacoustic, fluorescent tags).

Separation techniques:

Chromatography: Gas chromatography, liquid chromatography, supercritical fluids, Importance of column technology (packing, capillaries), Separation based on increasing number of factors (volatility, solubility, interactions with stationary phase, size, electrical field), Detection: simple vs. specific (gas and liquid), Detection as a means of further analysis (use of tags and coupling to IR and MS), Electrophoresis (plates and capillary) and use with DNA analysis.

Elemental analysis:

Mass spectrometry (electrical discharges).

Atomic spectroscopy: Atomic absorption, Atomic emission, and Atomic fluorescence.

Excitation and getting sample into gas phase (flames, electrical discharges, plasmas), Wavelength separation and resolution (dependence on technique), Detection of radiation (simultaneous/scanning, signal noise), Interpretation (errors due to molecular and ionic species, matrix effects, other interferences).

NMR spectroscopy:

Principle, Instrumentation, Factors affecting chemical shift, Spin-coupling, Applications.

Electroanalytical Methods:

Potentiometry & Voltammetry

Radiochemical Methods:

Elementary idea

X-ray analysis and electron spectroscopy (surface analysis):

Elementary idea

Suggested Readings:

1. D.A. Skoog, F.J. Holler & S. Crouch (ISBN 0-495-01201-7) *Principles of Instrumental Analysis*, Cengage Learning India Edition, 2007.
2. Willard, Merritt, Dean, Settle, *Instrumental Methods of Analysis*, 7th ed, IBH Book House, New Delhi.
3. Atkins, P.W & Paula, J.D. *Physical Chemistry*, 10th Ed., Oxford University Press (2014).
4. Kakkar, R. *Atomic and Molecular Spectroscopy: Concepts and Applications*. Cambridge University Press, 2015.
5. Castellan, G. W. *Physical Chemistry 4th Ed.*, Narosa (2004).
6. Banwell, C. N. & McCash, E. M. *Fundamentals of Molecular Spectroscopy 4th Ed.*
7. Smith, B.C. *Infrared Spectral Interpretations: A Systematic Approach*. CRC Press, 1998.
8. Moore, W.J., *Physical Chemistry* Orient Blackswan, 1999.

DSE2P: Instrumental Methods of Chemical Analysis (Lab)

Credits 02

Practical :

1. Safety Practices in the Chemistry Laboratory
2. Determination of the isoelectric pH of a protein.
3. Titration curve of an amino acid.
4. Determination of the void volume of a gel filtration column.
5. Determination of a Mixture of Cobalt and Nickel (UV/Vis spec.)
6. Study of Electronic Transitions in Organic Molecules (i.e., acetone in water)
7. IR Absorption Spectra (Study of Aldehydes and Ketones)
8. Determination of Calcium, Iron, and Copper in Food by Atomic Absorption
9. Quantitative Analysis of Mixtures by Gas Chromatography (i.e., chloroform and carbon tetrachloride)

10. Separation of Carbohydrates by HPLC
11. Determination of Caffeine in Beverages by HPLC
12. Potentiometric Titration of a Chloride-Iodide Mixture
13. Cyclic Voltammetry of the Ferrocyanide/ Ferricyanide Couple
14. Nuclear Magnetic Resonance
15. Use of fluorescence to do “presumptive tests” to identify blood or other body fluids.
16. Use of “presumptive tests” for anthrax or cocaine
17. Collection, preservation, and control of blood evidence being used for DNA testing
18. Use of capillary electrophoresis with laser fluorescence detection for nuclear DNA (Y chromosome only or multiple chromosome)

19. Use of sequencing for the analysis of mitochondrial DNA
20. Laboratory analysis to confirm anthrax or cocaine
21. Detection in the field and confirmation in the laboratory of flammable accelerants or explosives

22. Detection of illegal drugs or steroids in athletes
23. Detection of pollutants or illegal dumping
24. Fibre analysis

At least 10 experiments to be performed.

Suggested Readings:

1. Skoog, D.A. Holler F.J. & Nieman, T.A. *Principles of Instrumental Analysis*, Cengage Learning India Ed.
2. Willard, H.H., Merritt, L.L., Dean, J. & Settoe, F.A. *Instrumental Methods of Analysis*, 7th Ed. Wadsworth Publishing Company Ltd., Belmont, California, USA, 1988