

M.Phil & Ph.D. SYLLABUS
(With effect for students admitted during 2007 onwards)
**PAPER – I: RESEARCH METHODOLOGY AND INSTRUMENTAL
METHODS OF ANALYSIS**

CHAPTER – I: RESEARCH METHODOLOGY

Research methodology:

Choice of the research problem, collection of literature (Primary and secondary sources). Basic elements of writing a scientific paper and basic principles and techniques to be adopted in the preparation of M.Phil dissertation or Ph.D. thesis.

CHAPTER – 2: TREATMENT OF QUANTITATIVE DATA

Errors in Chemical Analysis: Definition of Terms; Systematic Errors;

Random Errors in Analysis:

The Nature of Random Errors; The Statistical Treatment of Random Error; The Standard Deviation of Computed Results; Methods for Reporting Computed Data:

Applications of Statistics to Data Treatment and Evaluation;

Confidence Limits; Statistical Aids to Hypothesis Testing; Detection of Gross Errors; Estimation of Detection limits; The Least-Squares Method for Deriving Calibration Plots; Quality Assurance and Control Charts. Significant figures.

CHAPTER – 3: GAS CHROMATOGRAPHY

Instruments for Gas-Liquid Chromatography; Principles of gas chromatography; Gas Chromatography Columns and Stationary Phases; Applications of Gas-Liquid Chromatography, Use of GC/MS to identify a Drug Metabolite in Blood, High-Speed Gas Chromatography; Gas-Solid Chromatography.

High-Performance Liquid Chromatography:

Instrumentation; Liquid Chromatography (LC)/Mass Spectrometry (MS) and LC/MS/MS; High-Performance Partition Chromatography; High-Performance Adsorption Chromatography; Ion-Exchange Chromatography; Size-Exclusion Chromatography, Buckyballs: The Chromatographic, Separation of Fullerenes; Affinity Chromatography; Chiral Chromatography; Comparison of High-Performance Liquid Chromatography and Gas Chromatography.

CHAPTER – 4: INFRARED AND RAMAN SPECTROSCOPY:

Theory of infra red absorption and Raman Spectroscopy, sample handling instrumentation (Basic Components) qualitative and quantitative applications. The Vibration-Rotation Spectrum of Carbon Monoxide; Breakdown of the Born-Oppenheimer Approximation; The Interaction of Rotations and Vibrations; The Vibrations of Polyatomic Molecules; The Influence of Rotation on the Spectra of Polyatomic Molecules; Analysis by Infra-red Techniques; Techniques and Instrumentation; Vibrational Raman Spectra; Polarization of Light and the Raman Effect; Structure Determination from Raman and Infra-red Spectroscopy; Techniques and Instrumentation.

CHAPTER – 5: RESONANCE SPECTROSCOPY

- (a) NMR: Theory of Nuclear magnetic Resonance, quantum description of NMR, classical description of NMR, Types of NMR spectra, environmental effects of NMR Spectra, the chemical shift, Block diagram of an NMR spectrometer, Applications of proton NMR in qualitative and quantitative analysis (in general).
- (b) ESR: Comparison between NMR and ESR, Types of substances with unpaired electrons, the any of ESR, Instrumentation presentation of the ESR spectrum. Hyper fine splitting, Determination of g-value, deviation of the value of g, line width, Applications of ESR spectroscopy ENDOR, ELDOR.

Suggested Books:

Chapter – 1:

- 1) Thesis and Assignment writing by Anderson.

Chapter – 2:

- 1) Fundamentals of Analytical Chemistry by Douglas A. Skoog, Donald M. West, F. James Holler, Harcourt College Publishers (7th Edition).

Chapter – 3:

- 1) Fundamentals of Analytical Chemistry by Douglas A. Skoog, Donald M. West, F. James Holler, Stanley R. Crouch THOMSON Brooks/Cole (8th Edition).

Chapter – 4:

- 1) Principles of instrumental analysis by D.A. Skoog Samnders college publishing Newyork 1985.
- 2) Fundamentals of molecular Spectroscopy by CN Banwell, TMH Edition, 1983.
- 3) Spectrometric identification of Organic compounds by Silverstein, bassler and Morril (John Wiley & sons) Newyork, 1981.
- 4) Coordination Chemistry by Burger.
- 5) Instrumental methods of analysis by Willard, Merritt and Dean.

Chapter – 5:

- 1) Spectroscopy by chatwal & Anand.

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OPTIONAL PAPER – I: INSTRUMENTAL METHODS OF ANALYSIS

CHAPTER – 1: SAMPLING, STANDARDIZATION & CALIBRATION

Analytical samples and Methods; Real Samples; sampling and sample Handling; obtaining a representative sample; sampling uncertainties, the Gross Sample; preparing a laboratory sample; number of laboratory sample; Automated sample handling; standardization and calibration; comparison with standards; external standard calibration, minimizing errors in analytical procedures, figures of merit for analytical methods, sensitivity and detection limit, linear dynamic Range; Quality Assurance of analytical results.

Ref Books:

1. Fundamentals of Analytical Chemistry 8th Edition, by Skoog, West, Holler & Crouch.

CHAPTER–2:INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY

Limitations of flame emission spectroscopy; Principles of plasma emission spectroscopy; Process of atomization and excitation; Plasma as an excitation source; Inductively coupled plasma source; ICP-AES instrumentation; Applications of plasma spectroscopy; Comparison of ICP-AES with AAS.

Ref Books:

1. Basic concepts of Analytical Chemistry by S.M. Khopkar.

CHAPTER – 3: ANALYSIS OF ENVIRONMENTAL SAMPLES

Air Pollution: Air Quality Standards; Sampling; *Monitoring:* Analysis of Carbon Monoxide, Analysis of Nitrogen Oxides (NO_x), Aromatic Hydrocarbons in Exhaust, Petrol and Air, Analysis of Particulate Matter, Spectrophotometric Analysis of Gaseous Air Pollutants.

Water Pollution: Waster Water Treatment; Domestic Waster Water Treatment; Drinking Water Supplies; Water Quality Parameters and Standards; Sampling; Preservation; *Monitoring Techniques and Methodology:* pH; Chemical Oxygen Demand (COD); Biochemical Oxygen Demand (BOD); Total Organic Carbon (TOC).

Ref: 1) Environmental Chemistry (Vth Edition), By A.K.DE, New Age International Publishers.

CHAPTER–4:PRINCIPLES AND PRACTICE OF SPECTROPHOTOMETRIC ANALYSIS

Introduction; colour and molecular structure; photometric laws; Double beam spectrophotometer and its functioning; sensitivity, selectivity and specificity of spectrophotometric methods; precision and accuracy of spectrophotometric methods; spectrophotometric reagents – Dathizone, Arsenaze III, Dithiocarbanate, 8-hydroxy quinoline; Derivative spectrophotometry and its merits; **Applications-** Determination of Mn(II) as MnO₄⁻, Fe(III) using thiocyanate, Cu(II) using salicylaldoxime, Pb(II) using dithizne; simultaneous determination of Mn(II) and Cr(VI).

- 1) Spectrophotometric determination of elements Z. Marczenko, Ellis Harwood ltd., Halsted press, Newyork, 1976.

- 2) Vogel's Text book of quantitative inorganic analysis; J. Basset, R.C. Denny, 5th Edition, Langman group, Lardan, G.H. Jeffery and J. Mendham.

CHAPTER – 5 :ELECTROCHEMICAL TECHNIQUES

Voltametry (Polarography)

Introduction Basic Principles D.C. Current polarography. Theoretical principles. Polarographic maxima. Half-wave potentials. Determination of formula and stability constant of metal ion complex. (Lingane method). Evaluation of quantitative result wave height – concentration plots. Internal standard (pilot ion) method and method of standard addition. Polarographs for classical d.c. polarography. Quantitative applications of polarography.

Inorganic and Organic analysis Determination of the half-wave potential of the cadmium ion in 1 M KCl solution. Determination of lead and copper in steel. Indirect determination of nitrate via orthonitrophenol. Determination of ascorbic acid in fruit juice. Cyclic voltametry stripping voltametry basic principles.

Coulometry: General discussion. Separation of Nickel and cobalt by coulometric analysis at controlled potential. Coulometry at constant current coulometric titrations.

Reference:

- 1) Vogel's & Text book of Quantitative chemical analysis (5th Edition).

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OPTIONAL PAPER – II: INORGANIC CHEMISTRY

CHAPTER – 1: BONDING IN METAL COMPLEXES:

Effective atomic number rule (EANR) and its applications, valence bond Theory (VBT)- inner and outer orbital complexes. Electronutrality principle and back bonding – strengths and shortcomings of the valence bond approach; Crystal Field Theory (CFT) – splitting of 'd' orbitals in tetra, oct, tetragonal and square planar complexes – Factors affecting the magnitude of Δ_0 . Calculation of crystal field stabilization Energy in tetra and octahedral complexes pairing Energy – applications of CFT with respect to color and magnetic properties of transition metal complexes. Jahn Teller Effect. Stability and Stability constants of complexes in Aqueous solutions and their thermodynamic origin. Their thermodynamic origin - Step – wise and overall stability constants and their relationship. Factors affecting the stability of the complex. Chelation and chelate effect Macrocyclic Effect; Stabilization of unusual oxidation states. Determination of stability constant of a complex using pH-metry, spectrophotometry and polarographic techniques.

CHAPTER – 2: MAGNETO CHEMISTRY

Magnetic properties of complexes; Fundamentals of magnetochemistry; Diamagnetism and paramagnetism; Magnetic susceptibility and magnetic moment; Fundamentals of applications of magnetochemistry in coordination chemistry; Factors determining paramagnetism. The 'spin-only' moment and its application in coordination chemistry; Orbital contribution, Investigation of the stereochemistry of complexes; The temperature dependence of magnetic susceptibility; Antiferromagnetism and its role in complex compounds; Effects of solvent on the magnetic properties of complexes. Diamagnetic-paramagnetic equilibria in solution; Diamagnetic-paramagnetic equilibria in solid complexes; Experimental methods; Gouy's method; Quincke's method; The magnet; Calibrating substances;

CHAPTER – 3: ELECTRONIC SPECTROSCOPY OF METAL COMPLEXES:

Energy levels in an atom, microstates – calculation of microstates; Spectroscopic states – coupling of orbital angular momenta and spin angular momenta; Spin – orbit coupling (R-S coupling) Deviation of term symbols for p^2 and d^2 configurations. Determination of ground state terms – Hund's Rules; Hole formulation principle.

Electronic spectra of octahedral and tetrahedral complexes. Nature of electronic spectrum; Laporte orbital and spin selection rules; Transforming spectroscopic terms into Mulliken symbols. Spectra of d^1 to d^9 metal ions/complexes. Orgel diagrams, Tanabe Sugano diagrams and comparison between these diagrams.

Electronic spectra of square pyramidal complexes; binuclear complexes having M-M multiple bonding and mixed valencies. Charge transfer ($L \rightarrow M$ and $M \rightarrow L$) spectra in inorganic compounds. Nature of electronic spectra of f block metal complexes.

References:

1. Concise Inorganic chemistry by J.D. Lee ELBS 4th Edition, 1994.
2. Inorganic Chemistry by Shriver and Atkins Oxford University Press 4th Edition, 1999.
3. Structural methods in Inorganic chemistry. E.A.V. Ebsworth, David W.H. Rankin and S. Craddock (Great Britain) ELBS, 1987.

CHAPTER – 4: MOSSBAUER AND NQR SPECTROSCOPY:

Mossbauer spectra of metal complexes: Fundamental principles; Mossbauer Spectrometer – Source and absorber, motion devices, detection, reference substances and calibration; Isomeric shift; quadrupole splitting; magnetic splitting, Mossbauer spectra of iron compounds and tin compounds. **Applications** – investigation of back bonding; investigation of ferrocene and its derivatives; investigation of dithio-carbonate complexes; investigations of spinel oxides containing iron; investigation of polynuclear tin complexes.

Nuclear Quadrupole Resource (NQR) spectroscopy:

Theory; energy levels and selection rules; Interaction of nuclear quadrupole with electromagnetic radiation; **applications** – Nature of chemical bond; study of chloromethanes, chloroacetyl chlorides and disubstituted benzenes; structural information about group III halides; study of charge transfer compounds.

Reference Books:

- 1) Coordination Chemistry: Experimental methods – K. burger, London Butterworth group, England, 1973.
- 2) Physical principles in Inorganic chemistry – R.S. Drago.
- 3) Spectroscopy – G.R. Chatwal and S.K. Anand, Himatay publishing House, 2002.

CHAPTER – 5: APPLICATIONS OF COORDINATION COMPOUNDS IN BIOLOGY AND MEDICINE:

Metal – Nucleic Acid Interactions: The Basics – Nucleic acid structures ‘fundamental interactions with nucleic acids; Fundamental reactions with nuclear acids; Binding of tris (phenanthroline) metal complexes with DNA. Techniques to monitor binding. Applications of different metal complexes that bind nucleic acids (spectroscopic probes – metal fort printing reagents conformational probes)

Metal complexes in Medicine: Metal deficiency and diseases; Toxic effects of metals; Metals used for diagnosis and chemo therapy. Gold complexes in the treatment of Rheumatoid arthritis; Anti cancer Drugs platinum ammine halide ; Metalloenes and gold complexes; platinum anticancer drugs – A case study of Bioinorganic chemistry of platinum anticancer Drugs; Design of new Inorganic anticancer Drugs;

Determination of antibacterial and anti-fungicidal activities of metal complexes.

References:

- 1) Bertini, H.B. Gray, S.J. Lippand and JS Valentine Bioinorganic Chemistry, Viva books pvt., Ltd., New Delhi, 1998.

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OPTIONAL PAPER – III: ORGANIC CHEMISTRY

CHAPTER- 1:¹³C AND 2D NMR SPECTROSCOPY:

¹³C and NMR Spectroscopy: CW and PFT techniques – Types of CMR spectra – uncoupled – proton decoupled – off – resonance decoupled (SFORD) – selectivity decoupled and gated decoupled spectra. ¹³C-chemical shifts – factors affecting the chemical shifts – Homonuclear (¹³C- ¹³CJ) and heteronuclear (¹³C – ¹H, ¹³C – ²HJ) couplings.

Introduction to 2D – NMR: Classification of 2D experiments – 2D-J-resolved spectroscopy – HOMO and HETERO – 2D – J- resolved spectra. Correlation spectroscopy (COSY) – HOMO – COSY, HETERO – COSY, 2D – INADEQUATE and NOESY.

CHAPTER – 2:REAGENTS IN ORGANIC SYNTHESIS

Lithium aluminium hydride – Sodium borohydride – Lithium dialkyl cuprate – Electrophilic sulphur and selenium reagents – lead tetra acetate – Dicyclohexyl carbodimide – Trimethyl silyl iodide – Lithium diisopropyl amide (LDA) – 2,3-dichloro-5,6-dicyano – p-benzoquinone(DDQ) & Wilkinson's catalyst.

CHAPTER – 3:DESIGN OF ORGANIC SYNTHESIS:

Terminology – Retrosynthetic analysis – Functional group inter conversion – Disconnection – Synthons – Protecting groups (alcohol, carbonyl and amino groups) – chemo selectivity – regioselectivity and stereoselectivity – linear and convergent synthesis. Use of disconnection approach in the synthesis of multistriatin, Juvabione, Longifoline and hexahelicene.

CHAPTER-4: REACTIONS OF SYNTHETIC IMPORTANCE AND SYNTHESIS OF BIOMOLECULES;

Reactions of synthetic importance: Sharpless epoxidation, Asymmetric aldol condensation, Robinson annulation, Stark enamine reaction, ene reaction and Simon – smith reaction.

Synthesis and structure of cholesterol – Reserpine, PGE₁, Taxal, oxytocin and rotenone.

CHAPTER-5: STRUCTURAL ELUCIDATION OF THE FOLLOWING COMPOUNDS BY IR, NMR AND MASS SPECTROSCOPY.

1-3 – dibromopropane, 2,4-dinitro-1-fluoro benzene; β-bromo styrene, p-Cl-Benzophenone, isobutyl alcohol, Benzyl methyl sulphide, Diethyl fumarate, cinnamaldehyde, Neopentyl amine, morpholine, 2,3,6-trichloro phenol and Hydro cinnamic acid.

Reference:

- 1) Spectroscopic identification of organic compounds by R.M.silverstein, G.C. Bassler and T.E. Morrill.
- 2) Spectroscopic identification of organic compounds by R.M.Silverstein and Webster.
- 3) NMR in chemistry – A multinuclear introduction by William Kemp.
- 4) ¹³C NMR for Organic chemists by G.C. Levy and G.L. Nelson.
- 5) Advanced Organic chemistry Part B by F.A. Carey and R.J. Sundberg.
- 6) Organic synthesis: The disconnection approach by Stuart Warren.
- 7) Guide Book to Organic synthesis by R.K. Mickie, D.M.Smith and R.A. Atken.
- 8) Text book of Organic chemistry Vol II by I.L.Finar.

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OPTIONAL PAPER – IV: PHYSICAL CHEMISTRY

CHAPTER – 1: THERMODYNAMICS

The partition function: Energy, heat capacity and the partition function. Separation of energy contributions. Translational partition function. Diatomic molecules electronic partition function. Rotational partition function, Vibrational partition function. Entropy and partition function, Sackur Tetrode equation. Entropy of monoatomic molecules.

Partial Molar properties: Physical significance of partial molar property. Partial molar free energy chemical potential. Determination of partial molar properties – Direct method; From apparent molar properties. Determination of partial molar volume from density measurements.

Reference:

- 1) Thermodynamics for chemists by Samuel Glastone.
- 2) Basic Thermodynamics – by Evelgn Guha.
- 3) An introduction to chemical Thermodynamcis by R.P. Rastogi & RR. Misra.
- 4) Statistical Thermodynamic by A.K. Gupta.

CHAPTER -2: REACTION DYNAMICS

Molecular dynamical calculations; The reaction $H + H_2$. The Reaction $Br + H_2$; more complex reactions; molecular beams, stripping and rebound mechanisms; state to state kinetics; Influence of Reactant Vibrational energy; Influence of reactant notational energy; spectroscopy of transition species.

Reference:

- 1) Chemical kinetics (3rd Edition) by Keith J. Laidler., Chapter 12 Pages 449 – 484.

CHAPTER – 3: ELECTROCHEMICAL TECHNIQUES

Voltametry (Polarography)

Introduction Basic Principles D.C. Current polarography. Theoretical principles. Polarographic maxima. Half-wave potentials. Determination of formula and stability constant of metal ion complex. (Lingane method). Evaluation of quantitative result wave height – concentration plots. Internal standard (pilot ion) method and method of standard addition. Polarographs for classical d.c. polarography. Quantitative applications of polarography.

Inorganic and Organic analysis Determination of the half-wave potential of the cadmium ion in 1 M KCl solution. Determination of lead and copper in steel. Determination of nitrobenzene in aniline. Determination of ascorbic acid in fruit juice. Cyclic voltametry stripping voltametry basic principles.

Coulometry: General discussion. Separation of Nickel and cobalt by coulometric analysis at controlled potential. Coulometry at constant current coulometric titrations.

Reference:

- 1) Vogel's & Text book of Quantitative chemical analysis (5th Edition).

CHAPTER – 4:UV-VISIBLE SPECTROPHOTOMETRY:

Beers Law and its limitations; Definition of terms; Transmittance, absorbance, molar absorptivity, absorptivity, sandells sensitivity. Ringbom plot and its significance. Relative concentration error in photometric methods. Optical path and working at a spectrophotometer source, monochromaters and detectors used in spectrophotometers.

Optimal conditions in the development of spectrophotometric method for the analysis an absorbing species. Selectivity and sensitivity of spectrophotometric methods.

Applications: Determination of a single absorbing species (Manganese, Iron, Molybdenum oxalate fluoride) simultaneous determination of two absorbing species (manganese and chromium). Pka of an acid base indicator. Determination of formula and stability constant of metal complex – jobs method, molar ratio method and slope ratio method.

Reference:

- 1) Instrumental methods of analysis Principles of illard, merit and dean.
- 2) Instrumental analysis skoog and west.

CHAPTER–5: STRUCTURAL ELUCIDATION OF THE FOLLOWING COMPOUNDS BY IR, NMR AND MASS SPECTROSCOPY.

1-3 – dibromopropane, 2,4-dinitro-1-fluoro benzene; β -bromo styrene, p-Cl-Benzophenone, isobutyl alcohol, Benzyl methyl sulphide, Diethyl fumarate, cinnamaldehyde, Neopentyl amine, morpholine, 2,3,6-trichloro phenol and Hydro cinnamic acid.

- 1) Spectroscopic identification of organic compounds by R.M.silverstein, G.C. Bassler and T.E. Morrill.
- 2) Spectroscopic identification of organic compounds by R.M.Silverstein and Webster.

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OPTIONAL PAPER – V: ANALYTICAL CHEMISTRY

CHAPTER – 1: SAMPLING, STANDARDIZATION & CALIBRATION;

Analytical samples and Methods; Real Samples; sampling and sample Handling; obtaining a representative sample; sampling uncertainties, the Gross Sample; preparing a laboratory sample; number of laboratory sample; Automated sample handling; standardization and calibration; comparison with standards; external standard calibration, minimizing errors in analytical procedures, figures of merit for analytical methods, sensitivity and detection limit, linear dynamic Range; Quality Assurance of analytical results.

Ref Books:

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**CHAPTER – 2: INDUCTIVELY COUPLED PLASMA-ATOMIC
EMISSION SPECTROSCOPY:**

Limitations of flame emission spectroscopy; Principles of plasma emission spectroscopy; Process of atomization and excitation; Plasma as an excitation source; Inductively coupled plasma source; ICP-AES instrumentation; Applications of plasma spectroscopy; Comparison of ICP-AES with AAS.

Ref Books:

1. Basic concepts of Analytical Chemistry by S.M. Khopkar.

CHAPTER – 3: ANALYSIS OF ENVIRONMENTAL SAMPLES:

(a) Water Pollution: Waster Water Treatment; Domestic Waster Water Treatment; Drinking Water Supplies; Water Quality Parameters and Standards; Sampling; Preservation; *Monitoring Techniques and Methodology*: pH; Chemical Oxygen Demand (COD); Biochemical Oxygen Demand (BOD); Total Organic Carbon (TOC).

(b) Toxicological Chemistry: Introduction Toxicology and Toxicological Chemistry; Kinetic Phase and Dynamic Phase; Teratogenesis, Mutagenesis, Carcinogenesis, Immune System Effects and Reproductive Effects; Health hazards; Toxicology of Inorganic & Organic compounds; Effects of toxic chemicals on enzymes, biochemical effects of As, Cd, Hg, Pb and oxides of sulphur and nitrogen.

Ref Books:

1. Environmental Chemistry (Vth Edition), By A.K.DE, New Age International Publishers.
2. Environmental Chemistry by Stanley E. Manahan (VI Edition).

CHAPTER–4:

(a) Analytical method development and validation:

Basic concepts and control; column selection; Buffer selection; Solvent selection; Type of detectors; Trouble shooting on separation; Definition of validation; validation parameters: Specificity; System suitability; LOD; LOQ; Linearity and range; Accuracy, robustness and ruggedness.

(b) Quality & quality assurance: Definition; Need of quality system; Benefits of quality system; Approaches in quality.

Ref Books:

- 1) Instrumentation to modern chromatography by L.R.Snyder and J.J. Kirkland (John Wiley and Sons, Inc) II Edition.
- 2) Analytical Method development and validation by Michael Swatz & Iras.Krull.
- 3) Analytical Instrumentation performance characteristic and quality by Graham Currell.

CHAPTER-5:

(a) Structural elucidation of the following compounds by IR, NMR, ESR, Mass spectroscopy.

Benzyl Alcohol; Diethyl malonate; P-chlorobenzophenone; n-propyl fumarate; 2-Bromo proionic acid; 1,3,5-Trimethyl Benzene; Phenyl Acetaldehyde; Methyl cyclohexane; Diethyl fumarate; Hydro cinnamic acid.

(b) Spectroscopic Interpretation and applications in Inorganic Chemistry:

ESR Analysis of Cu(I) & Cu(II) complexes and Hydroquinone; NMR Analysis of inorganic compounds (BrF₅ & SF₄); IR Analysis of Aldehydes & Ketones.

Ref Books:

- 1) Spectroscopy by H. Kaur.
- 2) Engineering Chemistry by Jain and Jain.
- 3) Spectroscopic identification of Organic compounds by Robert M. Silver Stein.