KRISHNA UNIVERSITY
MACHILIPATNAM – 521003 (A.P) INDIA
DEPARTMENT OF CHEMISTRY
Ph.D-M.Phil-Chemistry-Pre-PhD-Syllabus

PAPER-II: Organic Spectroscopy & Chromatography Techniques

UNIT-I: UV-Visible Spectroscopy: Brief review of the electromagnetic spectrum, UV-Visible range, energy-wavelength-colour relationships, Interaction of electromagnetic radiation (UV- Vis) with matter and its effects, Chromophores, Auxochromes, bathochromic, hypochromic, hypochromic, hyperchromic shifts. Calculation of λmax. Woodward-Hofmann rules for conjugated dienes and α,β-Unsaturated carbonyl compounds.

UNIT-II: IR Spectroscopy: Identification of functional groups, confirming the molecules with IR, modes of fundamental vibrations, estimating the purity of compound, fingerprint region.

UNIT-III: Mass Spectrometry: Basic principles and a brief outline of instrumentation. Ion formation and types, molecular ion, metastable ions, Fragmentation processes, Fragmentation patterns, nitrogen rule, identification of chloro and bromo compounds, Mc Lafferty rearrangement, Mass spectrum, its characteristics and representation.

UNIT-IV: NMR: Reference, Chemical shift, solvents used in NMR, D2O exchange, identification of nature of protons and number of protons on the particular chemical environment.

UNIT-V: Chromatography: Introduction & classification of chromatographic techniques. Principle, instrumentation and applications of different chromatographic techniques - TLC, HPLC, Column Chromatography, Ion exchange chromatography.

Reference Books:

1. Organic spectroscopy by Y.R Sharma.
2. Organic spectroscopy by William Kemp.
3. Spectroscopy of organic compounds by P.S.Kalsi.
4. Analytical Chemistry for Technicians, Fourth Edition John Kenkel |2013|.
5. Chromatography Concepts and Contrasts James M. Miller |2009|.

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PAPER-III: Organic Synthesis

UNIT-I: Purification and drying of chemicals and solvents: Purification and drying of Benzene, hexane, toluene, xylene, methanol, ethanol, chloroform, carbon tetrachloride, dichloromethane, acetone, diethyl ether, tetrahydrofuran, dioxane, DMSO, DMF, TFA. Distillation techniques, crystallization techniques, drying techniques.

Unit-II: Oxidations, Reductions and Organometallic Reagents: Oxidations: Introduction, preparation, properties and synthetic applications of Oxidizing Reagents. **Reductions:** Introduction, preparation, properties and synthetic applications of Reducing Reagents. **Organometallics:** Introduction, preparation, properties and synthetic applications of Organometallic Reagents.

UNIT-III: Organic Named Reactions: Aldol, Benzoin, Cannizzaro, Perkin, Stobbe, Dieckmann condensations, Claisen, Hofmann, Schmidt, Lossen, Curtius, Beckmann and Fries rearrangements, Reimer-Tiemann, Reformatsky, Diels Alder reactions, Friedel crafts, Wittig, Mannich, Vilsmeier reactions, Robinson annulations.

Unit-IV: Heterocyclic compounds: Introduction, Definition, nomenclature, classification: Heterocyclics with single heteroatom, three, four, five and six membered heterocyclics. Heterocyclic with more than one hetero atom.

Unit-V: Chemistry of Nanomaterials: Introduction, carbon nanotubes: structure of single and multi-walled carbon nanotubes, synthesis-solid and gaseous carbon source-based production techniques, synthesis with controlled orientation. Growth mechanism of carbon nano tubes-catalyst free growth, catalyst activated growth, properties-general, adsorption, electronic and optical, Mechanical and reactivity. Applications.

Reference Books :

1. Advances in Organic Reaction mechanism and structure J. March (McGrew Hill).
2. A Guide Book to Mechanism in Organic Chemistry" by P.Sykes.
3. Synthetic approaches in organic chemistry by R.K.Bansal (Narosa Publications).
4. Some modern methods of synthesis by Carruthers (Cambridge).
5. V.S. Muralidharan A. Subramania, Nanoscience and Technology, Ane Books.

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Paper -III Analytical Chemistry

UNIT-I

Quality control in Analytical Chemistry

Quality assurance and management systems: elements of quality assurance, quality assurance in design, development, meaning of quality and customer requirement of quality. Quality management system, ISO 9000 and ISO 14000 series- statistical process control, process control tools.

Good laboratory practices (GLP): Need for GLP, GLP organization and management, Brief outline of ICH guidelines on drug substances and products.

UNIT-II

Precipitation methods

Nucleation and Crystal growth, homogeneous and heterogeneous nucleation, solubility and particle size, completeness of precipitation, effect of excess precipitant, pH, complex formation, temperature, purity of precipitates. Theory of co-precipitation, mixed crystal formation by occlusion and entrapment, re-precipitation with examples, theory of post-precipitation, examples of post-precipitation.

UNIT-III

Analysis of Water

Types of water pollutants and their effects, Analytical methods for the determination of the following ions in water, anions like CO_3^{2-}, HCO_3^-, F^-, Cl^-, SO_4^{2-}, PO_4^{3-}, NO_3^-, NO_2^-, CN^-, and S^{2-}. Determination of Cations in water: Fe^{2+}, Fe^{3+}, Ca^{2+}, Mg^{2+}, Cr^{3+}, As^{5+}, Pb^{2+}, Hg^{2+},Cu^{2+}, Zn^{2+},Cd^{2+}, Co^{2+}. Determination of Dissolved oxygen (D.O), Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD), standards for drinking water.

UNIT-IV

Analysis of Air

Composition of pure air, classification of air pollutants, chemical analysis for the following. Primary pollutants: Carbon compounds - Carbon monoxide(CO) and Carbon dioxide(CO_2). Sulphur compounds- sulphur dioxide (SO_2), Sulphur trioxide (SO_3) Nitrogen compounds -

nitric oxide (NO) and nitrogen dioxide (NO_2).

Hydrocarbons - Aliphatic hydrocarbons and polycyclic aromatic hydrocarbons, Inorganic and Organic particulates. Secondary pollutants - ozone (O_3), peroxy acetyl nitrate (PAN), peroxy benzyl nitrate (PBN). Standards for ambient air quality.

UNIT-V

Decomposition techniques in analysis

Principle of decomposition and Dissolution. Difference between dissolution and decomposition. Decomposition of samples with acids - HCI, HF, HNO_3, H_2SO_4 and $HClO_4$. Decomposition of samples by fusion, Alkali Fusion- Na_2CO_3, NaOH, Acidic Fusion- Sodium Hydro Sulphate, Sodium Pyrosulphate, Oxidation Fusion-Na_2O_2, Sodium Chlorate, Reductive Fusion Na_2CO_3 +Na_4BO_4. Sintering, difference between sintering and fusion. Decomposition of samples by sintering with sodium peroxide, sodium carbonate

References books:

1. Quality Assurance and Good Laboratory Practices, Prof. Y. Anjaneyulu, In Now Publication,
   New York.
2. Quality Assurance in Analytical Chemistry - G.Kateman and F.W Pipers, John Wiley and Sons, New York.
3. Technical methods of analysis - Griffin, MC Graw Hill Book Co.
4. Chemical analysis - H.A Laitinan, Me Graw Hill Book Co.
5. Newer redox titrants - Berka, Zyka and Vulterin, Pergamon Press.
6. Volumetric Analysis, Vol III- 1.M Kolthoff and R. Belcher, Interscience Public, New York.
7. Vogel's Text Book of Inorganic Quantitative Analysis - J. Bassett et al, ELBS.
8. Analytical Chemistry, An Introduction, D.A. Skoog, D.M West and F.J Holler, Sanders College Publishing, New York.
9. An Introduction ISO 9000, ISO 1400 Series, Environmental Management. K.V.S.G. Murali Krishna.