COLLEGE ANALYTICAL CHEMISTRY

For Third Year B.Sc.

(As per Revised Syllabus)

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SYLLABUS

SEMESTER V (USCH504)

UNIT 1 INTRODUCTION TO QUALITY CONCEPTS, CHEMICAL CALCULATIONS AND SAMPLING

1.1 Quality in Analytical Chemistry [05L]

1.1.1 Concepts of Quality, Quality Control and Quality Assurance

1.1.2 Importance of Quality Concepts in Industry

1.1.3 Chemical Standards and Certified Reference Materials; Importance in Chemical Analysis Quality of Material: Various Grades of Laboratory Reagents

1.2 Chemical Calculations (Numericals and word problems are expected) [04L]

1.2.1 Inter Conversion of Various Concentration Units. (Conversion of Concentration from One Unit to Another Unit with Examples)

1.2.2 Percent Composition of Elements in Chemical Compounds

1.3 Sampling [06L]

1.3.1 Purpose, Significance and Difficulties Encountered in Sampling

1.3.2 Sampling of Solids: Sample Size – Bulk Ratio, Size to Weight Ratio, Multistage and Sequential Sampling, Size Reduction Methods, Sampling of Compact Solids, Equipments and Methods of Sampling of Compact Solids, Sampling of Particulate Solids, Methods and Equipments used for Sampling of Particulate Solids.

1.3.3 Sampling of Liquids: Homogeneous and Heterogeneous, Static and Flowing Liquids.

1.3.4 Sampling of Gases: Ambient and Stack Sampling: Apparatus and Methods for Sampling of Gases.

1.3.5 Collection, Preservation and Dissolution of the Sample.

UNIT 2 CLASSICAL METHODS OF ANALYSIS (TITRIMETRY)

2.1 Redox Titrations (Numerical and word problems are expected) [08L]

2.1.1 Introduction

2.1.2 Construction of the Titration Curves and Calculation of Esystem in aqueous medium in case of:

(1) One Electron System

(2) Multielectron System

2.1.3 Theory of Redox Indicators, Criteria for Selection of an Indicator Use of Diphenyl Amine and Ferroin as Redox Indicators

2.2 Complexometric Titrations [07L]

2.2.1 Introduction, Construction of Titration Curve

2.2.2 Use of EDTA as Titrant and Its Standardisation, Absolute and Conditional Formation Constants of Metal EDTA Complexes, Selectivity of EDTA as a Titrant,

Factors Enhancing Selectivity with Examples. Advantages and Limitations of EDTA as a Titrant.

2.2.3 Types of EDTA Titrations.
2.2.4 Metallochromic Indicators, Theory, Examples and Applications

UNIT 3 OPTICAL METHODS

3.1 Atomic Spectroscopy: Flame Emission Spectroscopy (FES) and Atomic Absorption Spectroscopy (AAS) [07L]

3.1.1 Introduction, Energy Level Diagrams, Atomic Spectra, Absorption and Emission Spectra
3.1.2 Flame Photometry – Principle, Instrumentation (Flame Atomizers, Types of Burners, Wavelength Selectors, Detectors)
3.1.3 Atomic Absorption Spectroscopy – Principle, Instrumentation (Source, Chopper, Flame and Electrothermal Atomiser)
3.1.4 Quantification Methods of FES and AAS – Calibration Curve Method, Standard Addition Method and Internal Standard Method.
3.1.5 Comparison between FES and AAS
3.1.6 Applications, Advantages and Limitations

3.2 Molecular Fluorescence and Phosphorescence Spectroscopy [04L]

3.2.1 Introduction and Principle
3.2.2 Relationship of Fluorescence Intensity with Concentration
3.2.3 Factors Affecting Fluorescence and Phosphorescence
3.2.4 Instrumentation and Applications
3.2.5 Comparison of Fluorimetry and Phosphorimetry
3.2.6 Comparison with Absorption Methods

3.3 Turbidimetry and Nephelometry [04L]

3.3.1 Introduction and Principle
3.3.2 Factors Affecting Scattering of Radiation: Concentration, Particle Size, Wavelength, Refractive Index
3.3.3 Instrumentation and Applications

UNIT 4 METHODS OF SEPARATION – I

4.1 Solvent Extraction [06L]

4.1.1 Factors Affecting Extraction: Chelation, Ion Pair Formation and Solvation
4.1.2 Graph of Percent Extraction versus pH. Concept of $[pH]_{1/2}$ and Its Significance (derivation not expected)
4.1.3 Craig's Counter Current Extraction: Principle, Apparatus and Applications
4.1.4 Solid Phase Extraction: Principle, Process and Applications with Special Reference to Water and Industrial Effluent Analysis.
4.1.5 Comparison of Solid Phase Extraction and Solvent Extraction.

4.2 High Performance Liquid Chromatography (HPLC) [06L]

4.2.1 Introduction and Principle
Instrumentation - Components with Their Significance: S
Degassing System, Pumps - (Reciprocating Pumps,
Syringe Type Pumps, Pneumatic Pumps, Advantages an
of Each Pump), Precolumn, Sample Injection System, I
Detectors (UV – Visible Detector, Refractive Index I
4.2.2 Qualitative and Quantitative Applications of HPLC

4.3 High Performance Thin Layer Chromatography (HPTLC) [03L]

4.3.1 Introduction and Principle, Stationary phase, Sample Application and Mobile Phase
4.3.2 Detectors
(a) Scanning Densitometer - Components.
Types of densitometer - Single beam and Double beam
(b) Fluorometric Detector
4.3.3 Advantages, Disadvantages and Applications
4.3.4 Comparison of TLC and HPTLC

SEMESTER VI (USCH604)

UNIT 1 ELECTRO ANALYTICAL TECHNIQUES

1.1 Polarography (Numerical and word problems are expected) [11L]

1.1.1 Difference between Potentiometry and Voltammetry, Polarizable and Non-polarizable Electrodes
1.1.2 Basic Principle of Polarography H Shaped Polarographic Cell, DME (Construction, Working, Advantages and Limitations)
1.1.3 DC Polarogram: Terms Involved - Residual Current, Diffusion Current, Limiting Current, Half-Wave Potential - Role and Selection of Supporting Electrolyte, Interference of Oxygen and Its Removal, Polarographic Maxima and Maxima Suppressors Qualitative Aspects of Polarography: Half Wave Potential $E_{1/2}$, Factors Affecting $E_{1/2}$
Quantitative Aspects of Polarography: Ilkovic Equations: Various Terms Involved in It (No derivation)
1.1.4 Quantification
(1) Wave Height – Concentration Plots (Working Plots/Calibration)
(2) Internal Standard (Pilot Ion) Method
(3) Standard Addition Method
1.1.5 Applications Advantages and Limitations

1.2 Amperometric Titrations [04L]

1.2.1 Principle, Rotating Platinum Electrode (Construction, Advantages and Limitations)
1.2.2 Titration Curves with Example
1.2.3 Advantages and Limitations

UNIT 2 METHODS OF SEPARATION - II

2.1 Gas Chromatography (Numerical and word problems are expected) [09L]

on, Principle, Theory and Terms Involved
ation: Block Diagram and Components, Types of Columns,
Phases in GSC and GLC, Detectors: TCD, FID, ECD
, Quantitative Analysis and Applications
n between GSC and GLC

romatography [06L]

on, Principle.
on Exchangers, Ideal Properties of Resin

2.2.3 Ion Exchange Equilibria and Mechanism, Selectivity Coefficient and Separation Factor Factors Affecting Separation of Ions

2.2.4 Ion Exchange Capacity and Its Determination for Cation and Anion Exchangers.

2.2.5 Applications of Ion Exchange Chromatography with Reference to Preparation of Demineralised Water, Separation of Amino Acids

UNIT 3 FOOD AND COSMETICS ANALYSIS

3.1 Introduction to Food Chemistry [10L]

3.1.1 Food Processing and Preservation:
Introduction, Need, Chemical Methods, Action of Chemicals (Sulphur Dioxide, Boric Acid, Sodium Benzoate, Acetic Acid, Sodium Chloride and Sugar) and pH Control
Physical Methods (Pasteurization and Irradiation)

3.1.2 Determination of Boric Acid by Titrimetry and Sodium Benzoate by HPLC.

3.1.3 Study and Analysis of Food Products and Detection of Adulterants

(1) Milk: Composition & Nutrients, Types of Milk (Fat Free, Organic and Lactose Milk)
Analysis of Milk for Lactose by Lane Eynon's Method

(2) Honey: Composition Analysis of Reducing Sugars in Honey by Coles Ferricyanide Method

(3) Tea: Composition, Types (Green Tea and Mixed Tea) Analysis of Tannin by Lowenthal's Method

(4) Coffee: Constituents and Composition, Role of Chicory
Analysis of caffeine by Bailey Andrew method

[05L]

3.2 Cosmetics

3.2.1 Introduction and Sensory Properties

3.2.2 Study of Cosmetic Products –

(1) Face Powder: Composition Estimation of Calcium and Magnesium by Complexometric Titration

(2) Lipstick: Constituents Ash Analysis for Water Soluble Salts: Borates, Carbonates and Zinc Oxide

(3) Deodorants and Antiperspirants: Constituents, Properties Estimation of Zinc by Gravimetry

UNIT 4 THERMAL METHODS AND ANALYTICAL METHOD VALIDATION [12L]

4.1 Thermal Methods

4.1.1 Introduction to Various Thermal Methods
(TGA, DTA and Thermometric Titration)

4.1.2 Thermogravimetric Analysis (TGA)
Instrumentation-block Diagram, Thermobalance (Basic Components: Balance, Furnace, Temperature Measurement and Control, Recorder)
Thermogram (TG Curve) for $CaC_2O_4 \cdot H_2O$ and $CuSO_4 \cdot 5H_2O$
Factors Affecting Thermogram-Instrumental Factors and Sample Characteristics
Applications: Determination of Drying and Ignition Temperature Range
Determination of Percent Composition of Binary Mixtures

4.1.3 Differential Thermal Analysis (DTA):
Principle, Instrumentation, and Reference Material Used
Differential Thermogram (DTA Curve) $CaC_2O_4 \cdot H_2O$ and $CuSO_4 \cdot 5H_2O$
Applications
Comparison between TGA and DTA.

4.1.4 Thermometric Titrations – Principle and Instrumentation
Thermometric Titrations of :
(1) HCl v/s NaOH
(2) Boric acid v/s NaOH
(3) Mixture of Ca^{+2} and Mg^{+2} v/s EDTA
(4) Zn^{+2} with Disodium Tartarate.

4.2 Analytical Method Validation [03L]

4.2.1 Introduction and Need for Validation of a Method

4.2.2 Validation Parameters: Specificity, Selectivity, Precision, Linearity, Accuracy and Robustness

Note: Concept of sensitivity is to be discussed for all techniques and instruments mentioned in the syllabus.