

# T.Y.B.Sc. CHEMISTRY (6 UNITS)

## SEMESTER V

### ANALYTICAL CHEMISTRY

COURSE CODE: USCH504

CREDITS: 02

LECTURES: 60

<b>UNIT I :INTRODUCTION TO QUALITY CONCEPTS,CHEMICAL CALCULATIONS AND SAMPLING (3 &amp; 6 UNITS)</b>			
<b>1.1</b>	<b>Quality in Analytical Chemistry</b>		<b>05 L</b>
	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	
<b>1.2</b>	<b>Chemical Calculations (Numericals and word problems are expected)</b>		<b>04 L</b>
	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	
<b>1.3</b>	<b>Sampling</b>		<b>06 L</b>
	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.	
<b>UNIT II : CLASSICAL METHODS OF ANALYSIS (TITRIMETRY) (3 &amp; 6 UNITS)</b>			
<b>2.1</b>	<b>Redox Titrations (Numerical and word Problems are expected)</b>		<b>08 L</b>
	2.1.1	Introduction	
	2.1.2	Construction of the titration curves and calculation of $E_{\text{system}}$ 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	
<b>2.2</b>	<b>Complexometric Titrations</b>		<b>07 L</b>
	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	
<b>UNIT III: OPTICAL METHODS(6 UNITS)</b>			
<b>3.1</b>	<b>Atomic Spectroscopy: Flame Emission spectroscopy(FES) and Atomic Absorption Spectroscopy(AAS)</b>		<b>07 L</b>
	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	
<b>3.2</b>	<b>Molecular Fluorescence and Phosphorescence Spectroscopy</b>		<b>04L</b>
	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	
<b>3.3</b>	<b>Turbidimetry and Nephelometry</b>		<b>04 L</b>
	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	
<b>UNIT IV: METHODS OF SEPARATION – I (6 UNITS)</b>			
<b>4.1</b>	<b>Solvent Extraction</b>		<b>06 L</b>
	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.	
<b>4.2</b>	<b>High Performance Liquid chromatography (HPLC)</b>		<b>06L</b>
	4.2.1	Introduction and Principle  Instrumentation- components with their significance: Solvent Reservoir, Degassing system, Pumps-(reciprocating pumps, screw driven- syringe type pumps, pneumatic pumps, advantages and disadvantages of each pump), Precolumn, Sample injection system, HPLC Columns, Detectors(UV – Visible detector, Refractive index detector)	
	4.2.2	Qualitative and Quantitative Applications of HPLC	

<b>4.3</b>	<b>High Performance Thin Layer Chromatography (HPTLC)</b>		<b>03 L</b>
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		

### REFERENCES

1.	3000 solved problems in Chemistry, David E. Goldberg, PhD., Schaums Outline	Unit/s: (1.2)
2.	A guide to Quality in Analytical Chemistry: An aid to accreditation, CITAC and EURACHEM, (2002),	Unit/s (1.1)
3.	A premier sampling solids, liquids and gases, Smith Patricia I, American statistical association and the society for industrial and applied mathematics, (2001)	Unit/s (1.3)
4.	Analytical Chemistry, Gary.D Christan, 5th edition	Unit/s (4.1,4.2,4.3)
5.	Analytical Chemistry Skoog, West ,Holler,7th Edition:	Unit/s (2.1)
6.	Analytical Chromatography, Gurdeep R Chatwal, Himalaya publication	Unit/s (4.1,4.2,4.3)
7.	Basic Concepts of Analytical Chemistry, by S M Khopkar, new Age International (p) Limited	Unit/s (4.1,4.2,4.3)
8.	Chemical methods of separation, J A Dean, Van Nostrand Reinhold, 1969	Unit/s (4.1,4.2,4.3)
9.	Fundamentals of Analytical Chemistry by Skoog and West , 8th Edition	Unit/s (4.1,4.2,4.3)
10.	Handbook of quality assurance for the analytical chemistry laboratory, 2ndEdn., James P. DuxVanNostr and Reinhold, 1990	Unit/s (1.1)
11.	High Performance Thin Layer Chromatography by Dr P.D. Sethi, CBS Publisher and Distribution	Unit/s(4.1,4.2,4.3)
12.	High Performance Thin Layer Chromatography in Food analysis, by Prem kumar, CBS Publisher and distributor	Unit/s (4.1,4.2,4.3)
13.	Instrumental methods of Analysis, by Dr Supriya S	Unit/s (4.1,4.2,4.3)

	Mahajan, Popular Prakashan Ltd	
14.	Instrumental methods Of Analysis, by Willard Merritt Dean, 7thEdition, CBS Publisher and distribution Pvt Ltd	Unit/s (3.1,3.2,3.3)
15.	Instrumental Methods of Chemical Analysis by B.K. Sharma Goel Publishing House	Unit/s (4.1,4.2,4.3)
16.	Principles of Instrumental Analysis , 5th Edition, By Skoog, Holler, Nieman	Unit/s (4.1,4.2,4.3)(3.1,3.2,3.3)
17.	Quality control and Quality assurance in Analytical Chemical Laboratory, Piotr Konieczka and Jacek Namiesnik, CRC press (2018)	Unit/s (1.1)
18.	Quality in the Analytical Chemistry Laboratory, Elizabeth Prichard, Neil T. Crosby, Florence Elizabeth Prichard, John Wiley and Sons, 1995	Unit/s (1.1)
19.	Solvent extraction and ion exchange, J Marcus and A. S. Kertes Wiley INC 1969	Unit/s (4.1,4.2,4.3)
20.	Thin Layer Chromatography, A LAB. Handbook, Egon Stahl, Springer International Student Edition	Unit/s (4.1,4.2,4.3)

## PRACTICALS

### SEMESTER V

#### ANALYTICAL CHEMISTRY

**COURSE CODE: USCHP13**

**CREDITS: 02**

<ol style="list-style-type: none"> <li>1. Spectrophotometric estimation of fluoride</li> <li>2 Estimation of magnesium content in Talcum powder by complexometry, using standardized solution of EDTA</li> <li>3 Determination of COD of water sample.</li> <li>4 To determine potassium content of a Fertilizer by Flame Photometry (Calibration curve method).</li> <li>5 To determine the amount of persulphate in the given sample solution by back titration with standard Fe (II) ammonium sulphate solution.</li> <li>6 To determine the amount of sulphate in given water sample turbidimetrically.</li> </ol>
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**Note: Calculation of percent error is expected for all the experiments.**

## REFERENCES

1.	Vogel's Textbook of Quantitative Chemical Analysis, 5thEdn., G. H. Jeffery, J Bassett, J Memdham and R C Denney, ELBS with Longmann (1989).
2.	Vogel's Textbook of Quantitative Chemical analysis, Sixth edition, J.Mendham et.al

### SEMESTER VI ANALYTICAL CHEMISTRY

**COURSE CODE: USCH604**

**CREDITS: 02**

**LECTURES: 60**

#### UNIT I: ELECTRO ANALYTICAL TECHNIQUES(3 & 6 UNITS)

<b>1.1</b>	<b>Polarography (Numerical and word problems are expected)</b>	<b>11L</b>
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	
<b>1.2</b>	<b>Amperometric Titrations</b>	<b>04L</b>
1.2.1	Principle, Rotating Platinum Electrode(Construction, advantages and limitations)	
1.2.2	Titration curves with example	
1.2.3	Advantages and limitations	
<b>UNIT II: METHODS OF SEPARATION - II (3 &amp; 6 UNITS)</b>		
<b>2.1</b>	<b>Gas Chromatography (Numerical and word problems are expected)</b>	<b>09 L</b>

	2.1.1	Introduction, Principle, Theory and terms involved	
	2.1.2	Instrumentation: Block diagram and components, types of columns, stationary phases in GSC and GLC, Detectors: TCD, FID, ECD	
	2.1.3	Qualitative, Quantitative analysis and applications	
	2.1.4	Comparison between GSC and GLC	
<b>2.2</b>	<b>Ion Exchange Chromatography</b>		<b>06 L</b>
	2.2.1	Introduction, Principle.	
	2.2.2	Types of Ion 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	
<b>UNIT III:FOOD AND COSMETICS ANALYSIS(6 UNITS)</b>			
<b>3.1</b>	<b>Introduction to food chemistry</b>		<b>10 L</b>
	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 <b>1) Milk:</b> Composition & nutrients, types of milk (fat free, organic and lactose milk) Analysis of milk for lactose by Lane Eynon's Method <b>2) Honey:</b> Composition	

	<p>Analysis of reducing sugars in honey by Coles Ferricyanide method</p> <p><b>3) Tea:</b></p> <p>Composition, types (green tea and mixed tea) Analysis of Tannin by Lowenthal's method</p> <p><b>4) Coffee:</b></p> <p>Constituents and composition, Role of Chicory Analysis of caffeine by Bailey Andrew method</p>	
<b>3.2</b>	<b>Cosmetics</b>	<b>05 L</b>
	3.2.1	Introduction and sensory properties
	3.2.2	<p>Study of cosmetic products –</p> <p><b>1) Face powder:</b></p> <p>Composition Estimation of calcium and magnesium by complexometric titration</p> <p><b>2) Lipstick:</b></p> <p>Constituents Ash analysis for water soluble salts: borates, carbonates and zinc oxide</p> <p><b>3) Deodorants and Antiperspirants:</b></p> <p>Constituents, properties Estimation of zinc by gravimetry</p>
<b>UNIT IV: THERMAL METHODS AND ANALYTICAL METHOD VALIDATION</b>		
<b>(6 UNITS)</b>		
<b>4.1</b>	<b>Thermal Methods</b>	<b>12 L</b>
	4.1.1	Introduction to various thermal methods (TGA, DTA and Thermometric titration)
	4.1.2	<p><b>Thermogravimetric Analysis(TGA)</b></p> <p>Instrumentation-block diagram,thermobalance (Basic components: balance, furnace, temperature measurement and control, recorder)</p> <p>Thermogram (TG curve)for<math>\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}</math> and <math>\text{CuSO}_4 \cdot 5\text{H}_2\text{O}</math> Factors affecting thermogram-Instrumental factors and Sample characteristics</p> <p>Applications:</p> <p>Determination of drying and ignition temperature range Determination of percent composition of binary mixtures</p>



		(Estimation of Calcium and Magnesium oxalate)	
	4.1.3	<b>Differential Thermal Analysis (DTA):</b> Principle, Instrumentation, and Reference material used Differential thermogram ( DTA curve) $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ Applications Comparison between TGA and DTA.	
	4.1.4	<b>Thermometric Titrations</b> – Principle and Instrumentation Thermometric titrations of : 1) HCl v/s NaOH 2) Boric acid v/s NaOH 3) Mixture of $\text{Ca}^{+2}$ and $\text{Mg}^{+2}$ v/s EDTA 4) $\text{Zn}^{+2}$ with Disodium Tartarate.	
<b>4.2</b>	<b>Analytical Method Validation</b>		<b>03L</b>
	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.**

#### REFERENCES

1.	An Advance Dairy chemistry, V 3, P. F. Fox, P. L. H. McSweeney Springer	Unit/s (3.1,3.2)
2.	Analysis of food and Beverages, George Charalanbous, Academic press 1978	Unit/s (3.1,3.2)
3.	Analytical Chemistry of Open Learning(ACOL), James W. Dodd & Kenneth H. Tonge	Unit/s (4.1,4.2)
4.	Analytical chemistry David Harvey The ,McGraw Hill Companies, Inc.	Unit/s (4.1,4.2)
5.	Analytical Chemistry, Gary.D Christan, 5th edition	Unit/s (2.1,2.2)
6.	Analytical chemistry, R. K. Dave.	Unit/s (2.1,2.2)

7.	Chemical methods of separation, J A Dean, Van Nostrand Reinhold, 1969	Unit/s (2.1,2.2)
8.	Egyankosh.ac.in/bitstream/123456789/43329/1/Unit-8	Unit/s (1.1,1.2,1.3)
9.	Food Analysis, Edited by S. Suzanne Nielsen, Springer	Unit/s (3.1,3.2)
10.	Food Analysis: Theory and practice, YeshajahuPomeranz, Clifton E. Meloan, Springer	Unit/s (3.1,3.2)
11.	Formulation and Function of cosmetics, Sa Jellineck	Unit/s (3.1,3.2)
12.	Fundamentals of Analytical Chemistry, D .A. Skoog and D. M. West and F. J. Holler Holt., Saunders 6th Edition (1992)	Unit/s (2.1,2.2)
13.	Government of India publications of food drug cosmetic act and rules.	Unit/s (3.1,3.2)
14.	Harry's Cosmetology, Longman scientific co.	Unit/s (3.1,3.2)
15.	High Performance Thin Layer Chromatography in Food analysis, by Prem kumar, CBS Publisher and distributor	Unit/s (3.1,3.2)
16.	Instrumental methods Of Analysis, by Willard Merritt Dean, 7thEdition, CBS Publisher and distribution Pvt Ltd	Unit/s (1.1,1.2,1.3) (4.1,4.2,4.3)
17.	Introduction to Polarography and Allied Techniques, By Kamala Zutshi, New Age International, 2006.	Unit/s (1.1,1.2,1.3)
18.	Modern cosmetics, E. Thomessen Wiley Inter science	Unit/s (3.1,3.2)
19.	Principles of Instrumental Analysis , 5th Edition, By Skoog, Holler, Nieman	Unit/s (4.1,4.2,4.3)
20.	Principles of Polarography by Jaroslav Heyrovský , Jaroslav Kůta, 1st Edition, Academic Press, eBook ISBN: 978148326478	Unit/s (1.1,1.2,1.3)
21.	Solvent extraction and ion exchange, J Marcus and A. S. Kertes Wiley INC 1969	Unit/s (2.1,2.2,)

**PRACTICALS**  
**SEMESTER VI**  
**ANALYTICAL CHEMISTRY**

**COURSE CODE: USCHP14**

**CREDITS: 02**

- 1 Estimation of Chromium in water sample spectrophotometrically by using Diphenyl carbazide.
- 2 Estimation of reducing sugar in honey by Willstatter method.
- 3 Estimation of  $Mg^{+2}$  &  $Zn^{+2}$  by anion exchange resin.  
using an anion exchange resin
- 4 Estimation of acetic acid in Vinegar sample by using Quinhydrone electrode potentiometrically.
- 5 Determination of phosphoric acid in cola sample pH metrically.

**Note: Calculation of percent error is expected for all the experiments.**

**References:**

1.	Vogel's Textbook of Quantitative Chemical Analysis, 5thEdn., G. H. Jeffery, J Bassett, J Memdham and R C Denney, ELBS with Longmann (1989).
2.	Vogel's Textbook of Quantitative Chemical analysis, Sixth edition, J.Mendham et.al
3.	The chemical analysis of food and food products III edition Morris Jacob
4.	The chemical analysis of food by David Pearson and Henry Edward