# AC\_11-5-2017

Item No.\_\_\_\_\_

# **UNIVERSITY OF MUMBAI**

# **Syllabus for Approval**

Sr. No.	Heading	Particulars			
1	Title of Course	S. Y. B. Sc. Chemistry			
		F. Y. B. Sc. Passed from this university (or with ATKT in any two courses at the F. Y. B. Sc. Level) or equivalent qualification from other universities as may have been allowed by the relevant			
2	Eligibility for Admission	ordinances of this university			
3	Passing marks	40%			
4	Ordinances/Regulations (if any)				
5	No. of Semesters	Тwo			
6	Level	U.G.			
7	Pattern	Semester			
8	Status	New			
9	To be implemented from Academic year	2017-2018			

Date : 05-5-2017 BoS Chairperson: Convener: Dr. Ravindra G. Deshmukh Signature: Dr. Anil V. Karnik

# **UNIVERSITY OF MUMBAI**

# **Essentials Elements of The Syllabus**

4						
1	Title of Course	Syllabus for two semester S. Y. B. Sc. course in chemistry				
_		course in chemistryUSCH301USCH302USCH303				
2	Couse Code	USCH301, USCH302, USCH303				
		USCH401, USCH402, USCH404 USCHP1 to USCHP6				
		USCHP1 to USCHP6				
3	Preamble	Attached				
4	Objective	<ul> <li>inquiry into the fundamental aspects of the various core areas of Chemistry.</li> <li>To make the learner proficient in analysing the various observations and chemical phenomena presented to him during the course.</li> <li>To make the learner capable of solving problems in the various units of this course</li> <li>To give the learner an opportunity to get hands on experience of the various concepts and processes in the various branches of chemistry</li> <li>To impart various skills of handling chemicals, reagents, apparatus, instruments and the care and safety aspects involved in such handling</li> <li>To make the learner capable of analysing and interpreting results of the experiments he conducts or performs</li> </ul>				
5	Eligibility	Pass F. Y. B. Sc.				
6	Fee Structure	As Per Guidelines issued from the				
		University				
7	No. of Lectures	9 lectures per week (three lectures per				
-		paper)				
8	No. of Practicals	9 periods per week (three periods per				
		paper)				
9	Duration of Course	Two Semester				

10	Notional Hanna	72 hours non noner non competer Theory			
10	Notional Hours	12 nours per paper per semester Theory			
		and 36 hours per paper per semester for			
		laboratory sessions			
11	No of students per batch	120 students per division (20 Students for			
	-	laboratory sessions)			
12	Selection	As per merit.			
13	Assessment	End of semester examination of 100			
		marks per paper for theory and 50			
		marks per paper for laboratory sessions			
14	Syllabus Detail	Attached			
15	Title of the Unit	As given in the Syllabus text			
16	Title of the Sub-unit	As given in the syllabus text.			
17	Semester wise Theory	As prescribed in the syllabus text			
18	Semester wise Practicals	As prescribed in the syllabus text.			
19	Question Paper Pattern	As prescribed by the Faculty of Science			
20	Scheme of evaluation of	N.A.			
	Project				
21	List of suggested reading	As Attached			
22	List of websites	As Attached			
23	List of You Tube videos	As attached			
24	List of MOOCs	As Attached			

#### REGULATIONS

#### 1. Preamble and objectives of the Course :

In the first two semseters of the six semester graduation program of B. Sc.(Chemistry) the learner was introduced to some basic aspects in the various core branches of chemistry like Physical Chemistry, Organic chemistry and Inorganic chemistry. Concepts about the structure of atom, distribution of electrons, Thermodynamics, Formation of organic compounds and basic ideas in reactivity of molecules in general and organic compounds in particular were introduced to the learner. He was made inquisitive about why and how should atoms combine to give molecules or ions. The non-orbital approach to appreciating the shapes of polyatomic species in general and molecules in particular.

The story of chemistry is taken further in the coming two semesters of the second year of the B. Sc. (Chemistry) Program. However it is also realised that some students opting for the course on Chemistry may not continue with the subject subsequently as such the syllabus is designed to retain the interest of the serious learner of chemistry as well as be helpful to non-chemistry learners. With such students who would want to pursue other branches of science but would want to acquire a basic appreciation and experience of chemistry a separate paper (Paper-III) is designed. This paper along with the laboratory session unit that goes with it deals with the basics of chemical analysis, separating components from a given sample, basic concepts like pH, experimental techniques like Titrimetry, Gravimetry, using instruments to carry out analysis, the various techniques like chromatography, electrophoresis, Instrumentation in general is felt to be of interest to learners of various branches like physics, botany, zoology, and microbiology.

The major objectives of B.Sc. Chemistry course are

• To infuse in the learner a spirit of inquiry into the fundamental aspects of the various core areas of Chemistry.

- To make the learner proficient in analysing the various observations and chemical phenomena presented to him during the course.
- To make the learner capable of solving problems in the various units of this course
- To give the learner an opportunity to get hands on experience of the various concepts and processes in the various branches of chemistry
- To impart various skills of handling chemicals, reagents, apparatus, instruments and the care and safety aspects involved in such handling
- To make the learner capable of analysing and interpreting results of the experiments he conducts or performs
- To make the learner capable of acquiring or pursuing a source of livelihood like jobs in chemical industry
- To arouse the interest to pursue higher levels of learning in chemistry,
- 2. Condition for Admission

A candidate who has passed the F.Y.B.Sc. of Mumbai University or an examination of some other university accepted by the syndicate as equivalent there to with Chemistry, Physics, Maths, Botany, Zoology or Life Science shall be eligible for admission into S.Y.B.Sc., course in Chemistry.

То

- 3. Duration of the Course: one year
- 4. Course of study:

# Draft copy of the proposed revised syllabus for Choice Based Credit System S.Y.B.Sc. Chemistry To be implemented from the Academic year 2017-2018

For the subject of chemistry there shall be three papers for 45 lectures each comprising of three units of 15 L each.

#### Semester-III

1.	Paper-I (General Chemistry) Unit-I Physical Chemistry
	Unit-II Inorganic Chemistry
	Unit-III Organic Chemistry.
2.	Paper-II (General Chemistry) Unit-I Physical Chemistry
	Unit-II Inorganic Chemistry
	Unit-III Organic Chemistry.
3.	Paper III Basics of Analytical Chemistry

#### Semester-IV

1.	Paper-I (General Chemistry) U	Jnit-I	Physical Chemistry
	1	Unit-I	I Inorganic Chemistry
	1	Unit-I	II Organic Chemistry.
2.	Paper-II (General Chemistry) U	Unit-I	Physical Chemistry
	I	Unit-I	I Inorganic Chemistry
	1	Unit-I	II Organic Chemistry.
	E	Basics	of Analytical Chemistry
-			

3. Paper III

# Choice Based Credit System S. Y. B. Sc. Chemistry Syllabus To be implemented from the Academic year 2017-2018

# Course Content Semester III

<b>Course Code</b>	Unit	Topics	Credits	L/Week
USCH301	Ι	Chemical Thermodynamics-II,	2	1
		Electrochemistry		
	II			1
		Chemical Bonding		
	III	Reactions and reactivity of halogenated		1
		hydrocarbons, alcohols, phenols and epoxides		
USCH302	Ι	Chemical Kinetics-II, Solutions	2	1
	II	Selected topics on p block elements		1
	III	Carbonyl Compounds		1
USCH303	Ι	Intorduction to Analytical Chemistry and Statistical	2	1
		Treatment of analytical data-I		
	II	Classical Methods of Analysis.		1
	III	Instrumental Methods-I		1
USCHP1		Chemistry Practicals I	1	3
USCHP2		Chemistry Practicals II	1	3
USCHP3		Chemistry Practicals III	1	3

# Semester IV

Course Code	Unit	Topics	Credits	L/Week
USCH401	Ι	Electrochemistry-II, Phase Equilibria		1
	II	Comparative Chemistry of the transition metals &		1
		Coordination Chemistry		
	III	Carboxylic acids and their derivatives, Sulphonic acids		1
USCH402	Ι	Solid state, Catalysis	2	1
	II	Ions in aqeous medium & Uses and Environmental		1
		Chemistry of volatile Oxides and oxo-acids		
	III	Amines, Diazonium salts, Heterocyclic compounds		1
USCH403	Ι	Separation Techniques in Analytical Chemistry	2	1
	II	Instrumental Methods-II		1
	III	Statistical Treatment of analytical dataII		1
USCHP4		Chemistry Practicals I	1	3
USCHP5		Chemistry Practicals II	1	3
USCHP6		Chemistry Practicals III	1	3

# Semester III Paper I Theory: 45 Lectures

#### **Unit I:Physical Chemistry**

#### 1.1 Chemical Thermodynamics-II(8L)

1.1.1 Free Energy Functions: Helmholtz Free Energy, Gibb's Free Energy, Variation of Gibb's

free energy with Pressure and Temperature.

1.1.2 Gibbs-Helmholtz equation, van't Hoff reaction isotherm and van't Hoff reaction isochore.

(Numericals expected).

1.1.3 Thermodynamics of Open System: Partial Molal Properties, Chemical Potential and its variation with Pressure and Temperature, Gibb's Duhem equation.

1.1.4 Concept of Fugacity and Activity

#### **1.2 Electrochemistry: (7L)**

1.2.1 Conductivity, equivalent and molar conductivity and their variation with dilution for weak and strong electrolytes.

1.2.2 Kohlrausch law of independent migration of ions.

1.2.3 Applications of conductance measurements: determination of degree of ionization and ionization constant of weak electrolyte, solubility and solubility product of sparingly soluble salts, ionic product of water. (Numericals expected).

1.2.4 Transference number and its experimental determination using Moving boundary method. (Numericals expected). Factors affecting transference number.

#### Unit-II

#### **Chemical Bonding**

#### 2.1 Non-Directional Bonding

- 2.1.1 Ionic Bond: Conditions for the Formation of Ionic Bond.
- 2.1.2 Types of Ionic Crystals
- 2.1.3 Radius Ratio Rules
- 2.1.4 Lattice Energy, Borne-Lande Equation
- 2.1.5 Kapustinski Equation
- 2.1.6 Born-Haber Cycle and its Application

#### 2.2. Directional Bonding: Orbital Approach.

2.2.1 Covalent Bonding The Valence Bond Theory- Introduction and basic tenets.

# (6L)

(4L)

- 2.2.2 Interaction between two hydrogen atoms and the Potential energy diagram of the resultant system.
- 2.2.3 Corrections applied to the system of two hydrogen atoms- Formation of  $H_2$
- 2.2.4 Homonuclear diatomic molecules from He<sub>2</sub> to Ne<sub>2</sub>
- 2.2.5 Resonance and the concept of Formal Charge; Rules for Resonance or Canonical structures.
- 2.2.6 Bonding in Polyatomic Species: The role of Hybridization. And types of hybrid orbitals-*sp*,  $sp^2$ ,  $sp^3$ ,  $sp^3d$ ,  $sp^2d^2and sp^2d sp^3d^2$ .
- 2.2.7 Equivalent and Non-Equivalent hybrid orbitals
- 2.2.8 Contribution of a given atomic orbital to the hybrid orbitals (with reference to  $sp^3$  hybridisation as in CH<sub>4</sub>, NH<sub>3</sub> and H<sub>2</sub>O and series like NH<sub>3</sub>, PH<sub>3</sub>, AsH<sub>3</sub>, BiH<sub>3</sub>)

#### 2.3 Molecular Orbital Theory (5L)

- 2.3.1. Comparing Atomic Orbitals and Molecular Orbitals.
- 2.3.2. Linear combination of atomic orbitals. to give molecular orbitals LCAO-MO approach for diatomic homonuclear molecules).
- 2.3.4. Wave mechanical treatment for molecular orbitals  $(H_2^+ \text{ and } H_2)$
- 2.3.4 Molecular orbital Theory and Bond Order and magnetic property: with reference to  $O_2, O_2^+ O_2^-, O_2^{-2-}$

(Problems and numerical problems expected wherever possible)

#### **Unit III: Organic Chemistry**

#### 3.1.1. Reactions and reactivity of halogenated hydrocarbons: [4L]

- 3.1.1.**Alkyl halides:** Nucleophilic substitution reactions:  $S_N1$ ,  $S_N2$  and  $S_Ni$  mechanisms with stereochemical aspects and factors affecting nucleophilic substitution reactionsnature of substrate, solvent, nucleophilic reagent and leaving group.
- 3.1.2. Aryl halides: Reactivity of aryl halides towards nucleophilic substitution reactions. Nucleophilic aromatic substitution  $(S_NAr)$  addition-elimination mechanism and benzyne mechanism.

#### 3.1.2. Organomagnesium and organolithium compounds: [3L]

Nomenclature, nature, type and reactivity of carbon-metal bond. Preparation using alkyl / aryl halide. Structure, stability and reactions with compounds containing acidic hydrogen, carbonyl compounds, CO2, cyanides and epoxides.

#### 3.2 Alcohols, phenols and epoxides: [8L]

- 3.2.1.Alcohols: Nomenclature, Preparation: Hydration of alkenes, hydrolysis of alkyl halides, reduction of aldehydes and ketones, using Grignard reagent. Properties: Hydrogen bonding, types and effect of hydrogen bonding on different properties. Acidity of alcohols, Reactions of alcohols
- 3.2.2.**Phenols: Pr**eparation, physical properties and acidic character. Comparative acidic strengths of alcohols and phenols, resonance stabilization of phenoxide ion. Reactions of phenols.
- 3.2.3.**Epoxides:** Nomenclature, methods of preparation and reactions of epoxides: reactivity, ring opening reactions by nucleophiles (a) In acidic conditions: hydrolysis, reaction with halogen halide, alcohol, hydrogen cyanide. (b) In neutral or basic conditions: ammonia, amines, Grignard reagents, alkoxides.

## Semester III Paper II

## **Unit I: Physical Chemistry**

## 1.1 Chemical Kinetics-II (7L)

1.1.1 Types of Complex Chemical reactions: Reversible or opposing, consecutive and parallel reactions (No derivations, only examples expected ),

Thermal chain reactions: H. and Br. reaction. (only steps involved, no kinetic expression expected).

1.1.2 Effect of temperature on the rate of reaction, Arrhenius equation, Concept of energy of activation (Ea). (Numericals expected).

1.1.3 Theories of reaction rates: Collision theory and activated complex theory of bimolecular reactions. Comparison between the two theories

(Qualitative treatment only)

## 1.2 Solutions: (8 L)

1.2.1 Thermodynamics of ideal solutions: Ideal solutions and Raoult's law, deviations from Raoult's law–non-ideal solutions. Vapour pressure-composition and temperature -composition curves of ideal and non-ideal solutions. Distillation of solutions.Lever rule.Azeotropes.

1.2.2 Partial miscibility of liquids: Critical solution temperature; effect of impurity on partial miscibility of liquids with respect to Phenol-Water , Triethanolamine – Water and Nicotine – Water systems

1.2.3 Immiscibility of liquids- Principle of steam distillation.

1.2.4 Nernst distribution law and its applications, solvent extraction.

# Unit-II

## **2.** Selected topics on p block elements (15L)

#### 2.1 Chemistry of Boron compounds

- 2.1.1 Electron deficient compounds BH<sub>3</sub>, BF<sub>3</sub>, BCl<sub>3</sub> with respect to Lewis acidity and applications.
- 2.1.2 Preparation of simple boranes like diborane and tetraborane.
- 2.1.3 Structure and bonding in diborane and tetraborane (2e-3c bonds)
- 2.1.4 Synthesis of Borax.

#### 2.2 Chemistry of Silicon and Germanium

- 2.2.1 Silicon compounds: Occurrence, Structure and inertness of SiO<sub>2</sub>
- 2.2.2 Preparation of structure of SiCl<sub>4</sub>
- 2.2.3 Occurrence and extraction of Germanium
- 2.2.4 Preparation of extra pure Silicon and Germanium

#### 2.3 Chemistry of Nitrogen family

- 2.3.1 Trends in chemical reactivity Formation of hydrides, halides, oxides with special reference to oxides of nitrogen.
- 2.3.2 Oxides of nitrogen with respect to preparation and structure of NO, NO<sub>2</sub>,  $N_2O$  and  $N_2O_4$ .
- 2.3.3 Synthesis of ammonia by Bosch Haber process.

### **Unit III: Organic Chemistry**

#### Carbonyl Compounds: [15L]

- 3.1 Nomenclature of aliphatic, alicyclic and aromatic carbonyl compounds. Structure, reactivity of aldehydes and ketones and methods of preparation; Oxidation of primary and secondary alcohols using PCC, hydration of alkynes, action of Grignard reagent on esters, Rosenmund reduction, Gattermann Koch formylation and Friedel Craft acylation of arenes
- 3.2 General mechanism of nucleophilic addition, and acid catalyzed nucleophilic addition reactions.
- 3.3 Reactions of aldehydes and ketones with NaHSO<sub>3</sub>, HCN, RMgX, alcohol, amine, phenyl hydrazine, 2,4-Dinitrophenyl hydrazine, LiAlH<sub>4</sub> and NaBH<sub>4</sub>.
- 3.4 Mechanisms of following reactions: Benzoin condensation, Knoevenagel condensation, Claisen-Schmidt and Cannizzaro reaction.
- 3.5 Keto-enol tautomerism: Mechanism of acid and base catalysed enolization
- 3.6 Active methylene compounds: Acetylacetone, ethyl acetoacetate diethyl malonate, stabilised enols. Reactions of Acetylacetone and ethyl acetoacetate (alkylation, conversion to ketone, mono- and dicarboxylic acid)

# Semester IV Paper I

#### **Unit I: Physical Chemistry**

#### 1.1Electrochemistry-II: (8 L)

- 1.1.1 Electrochemical conventions, Reversible and irreversible cells.
- 1.1.2 Nernst equation and its importance, Types of electrodes, Standard electrode potential, Electrochemical series (Numericals expected).
- 1.1.3 Thermodynamics of a reversible cell, calculation of thermodynamic properties:  $\Delta G$ ,  $\Delta H$  and  $\Delta S$  from EMF data. (Numericals expected)
- 1.1.4 Calculation of equilibrium constant from EMF data. (Numericals expected)
- 1.1.5 Concentration cells with transference and without transference. Liquid junction potential and salt bridge.
- 1.1.6 pH determination using hydrogen electrode and quinhydrone electrode.(Numericals expected)

#### **1.2** Phase Equilibria: (7L)

- 1.2.1 Phases, components and degrees of freedom of a system, criteria of phase equilibrium. Gibbs Phase Rule and its thermodynamic derivation.
- 1.2.2 Derivation of Clausius Clapeyron equation and its importance in phase equilibria. (numericals expected)
- 1.2.3 Phase diagrams of one-component systems (water and sulphur).
- 1.2.4 Two component systems involving eutectics, congruent and incongruent melting points (lead-silver system).

#### Unit-II

#### 2.1 Comparative Chemistry of the transition metals (9 L)

- **2.1.1** Position in the periodic table; Natural occurrence principal ores and minerals;
- **2.1.2** Significance of special stability of  $d^0$ ,  $d^5$  and  $d^{10}$  leading to variable oxidation states; Unusual oxidation states and their stabilities in aqueous solutions (with special reference to vanadium, and chromium.)

- **2.1.3** Origin of colour for transition metals and their compounds: such as reflectivity, surface coatings, particle size, packing density for metals and nature of d-orbitals, number of electrons in the d-orbitals, geometry, and ability for charge transfer).
- 2.1.4 Magnetic properties of transition metal compounds: Origin of magnetism-spin and orbital motion of electrons; equation for spin only and spin-orbital magnetism in terms of Bohr magnetons (No derivation of relevant equations expected); Reasons for quenching of orbital moments.
- **2.1.5** Chemistry of Titanium and vanadium: properties of Oxides and chlorides; use in titrimetric analysis
- **2.1.6** Qualitative tests for transition metal ions: General considerations in devising tests (with reference to Chromium, Manganese, iron, Cobalt Nickel and Copper)

## 2.2 Coordination Chemistry : (6 L)

v.

## 2.2.1 Introduction to Chemistry of Coordination Compounds

- i. Historical perspectives: Early ideas on coordination compounds
- ii. Basic terms and nomenclature.
- iii. Types of ligands
- iv. Isomerism :General Types with special reference to stereoisomerism of coordination compounds (C.N=6)
  - Evidence for the formation of coordination compounds,

## 2.2.2. Theories of coordination compounds

- i. Werner's Theory of coordination compounds,
- ii. Effective atomic number rule.
- iii. Eighteen electron Rule

## 2.2.3. Nature of the Metal-Ligand Bond:

- i. Valence Bond Theory; Hybridisation of the central metal orbitals-sp<sup>3</sup>,  $sd^{3}/d^{3}s sp^{3}d^{2}/d^{2}sp^{3}$ ,  $sp^{2}d$ ,
- ii. Inner and outer orbital complexes of .(suitable examples of Mn(II) Fe(II),Fe(III),Co(II)/Co(III),Ni(II), Cu(II) Zn(II) complexes with ligands like aqua, ammonia CN<sup>-</sup> and halides may be used)
- iii. Limitations of V.B.T
- **2.2.4.** Application of coordination compounds.

## Unit III: Organic Chemistry

## 3.1 Carboxylic Acids and their Derivatives :(11 Lectures)

3.1.1. Nomenclature, structure and physical properties, acidity of carboxylic acids, effects of substituents on acid strength of aliphatic and aromatic carboxylic acids.

3.1.2. Preparation of carboxylic acids: oxidation of alcohols and alkyl benzene, carbonation of Grignard and hydrolysis of nitriles.

3.1.3. Reactions: Acidity, salt formation, decarboxylation, Reduction of carboxylic acids with LiAlH<sub>4</sub>, diborane, Hell-Volhard-Zelinsky reaction, Conversion of carboxylic acid to acid chlorides, esters, amides and acid anhydrides and their relative reactivity.

3.1.4. Mechanism of nucleophilic acyl substitution and acid-catalysed nucleophilic acyl substitution. Interconversion of acid derivatives by nucleophilic acyl substitution.3.1.5. Mechanism of Claisen condensation and Dieckmann condensation.

#### **3.2 Sulphonic acids: [4L]**

Nomenclature, preparation of aromatic sulphonic acids by sulphonation of benzene (with mechanism), toluene and naphthalene, Reactions: Acidity of arene sulfonic acid, Comparative acidity of carboxylic acid and sulfonic acids. Salt formation, desulphonation.Reaction with alcohol, phosphorous pentachloride, IPSO substitution.

# Semester IV Paper II

#### **Unit I: Physical Chemistry**

#### 1.1 Solid State: (7L)

- 1.1.1 Recapitulation of laws of crystallography and types of crystals
- 1.1.2 Characteristics of simple cubic, face centered cubic and body centered cubic systems, interplanar distance in cubic lattice (only expression for ratio of interplanar distances are expected)
- 1.1.3 Use of X-rays in the study of crystal structure, Bragg's equation (derivation expected), X-rays diffraction method of studying crystal lattice structure, structure of NaCl and KCl. Determination of Avogadro's number (Numericals expected)

#### 1.2 Catalysis: (8 L)

- 1.2.1 Types of catalysis, catalytic activity, specificity and selectivity, inhibitors, catalyst poisoning and deactivation
- 1.2.2 Mechanisms and kinetics of acid-base catalyzed reactions, effect of pH.
- 1.2.3 Mechanisms and kinetics of enzyme catalyzed reactions (Michaelis-Menten equation)
  - 1.2.4 Effect of particle size and efficiency of nanoparticles as catalyst. Unit-II

#### 2 Ions in aqeous medium

- 2.1. Acidity of Cations and Basicity of Anions
  - i. Hydration of Cations; Hydrolysis of Cations predicting degree of hydrolysis of Cations-effect of Charge and Radious.
  - ii. Latimer Equation. Relationship between pKa, acidity and  $z^2/r$  ratios of metal ions graphical Presentation
  - iii. Classification of cations on the basis of acidity category Non acidic, Moderately acidic, strongly acidic, very strongly acidic with pKa values range and examples
  - iv. Hydration of Anions; Effect of Charge and Radius; Hydration of anionsconcept, diagram classification on the basis of basicity

## 2.2. Uses and Environmental Chemistry of volatile Oxides and oxo-acids

- i. Physical properties of concentrated oxo-acids like sulfuric, Nitric and Phosphoric acid
- ii. Uses and environments aspects of these acids

## **Unit III: Organic Chemistry**

#### Nitrogen containing compounds and heterocyclic compounds:

3.1 Amines: Nomenclature, effect of substituent on basicity of aliphatic and aromatic amines;

- 3.1.1. Preparation: Reduction of aromaticnitro compounds using catalytic hydrogenation, chemical reduction using Fe-HCI, Sn-HCl, Zn-acetic acid, reduction of nitriles, ammonolysis of halides, reductive amination, Hofmann bromamide reaction.
- 3.1.2. Reactions- Salt Formation, N-acylation, N-alkylation, Hofmann's exhaustive methylation (HEM),Hofmann-elimination reaction, reaction with nitrous acid, carbylamine reaction, Electrophilic substitution in aromatic amines: bromination, nitration and sulphonation.

#### **3.2 Diazonium Salts: (7 Lectures)**

Preparation and their reactions/synthetic application - Sandmeyer reaction, Gattermann reaction, Gomberg reaction, Replacement of diazo group by -H,-OH. Azo coupling with phenols, naphthols and aromatic amines, reduction of diazonium salt to aryl hydrazine and hydroazobenzene

#### 3.3 Heterocyclic Compounds: (8 Lectures)

- 3.3.1. Classification, nomenclature, electronic structure, aromaticity in 5-numbered and 6-membered rings containing one heteroatom;
- 3.3.2. Synthesis of Furan, Pyrrole (Paal-Knorr synthesis, Knorr pyrrole synthesis, and Hantzsch synthesis), Thiophene, Pyridine (Hantzsch synthesis),
- 3.3.3. Reactivity of furan, pyrrole and thiophene towards electrophilic substitution reactions on the basis of stability of intermediate and of pyridine on the basis of electron distribution. Reactivity of pyridine towards nucleophilic substitution on the basis of electron distribution.
- 3.3.4. Reactions of furan, pyrrole and thiophene: halogenation, nitration, sulphonation, Vilsmeier-Haack reaction, Friedel-Crafts reaction. Furan: Diels-Alder reaction, Ring opening. Pyrrole: Acidity and basicity of pyrrole. Comparison of basicity of pyrrole and pyrrolidine.
- 3.3.5. Pyridine: Basicity. Comparison of basicity of pyridine, pyrrole and piperidine.Sulphonation of pyridine (with and without catalyst), reduction and action of sodamide (Chichibabin reaction).

# Semester III Chemistry Practicals:

#### **Unit I: Physical Chemistry**

- 1. To verify Ostwald's dilution law for weak acid conductometrically.
- 2. To determine dissociation constant of weak acid conductometrically.
- 3. To determine the critical solution temperature (CST) of phenol WaterSystem.
- 4. Determination of energy of activation of acid catalyzed hydrolysis of methyl acetate.
- 5. To investigate the reaction between  $K_2S_2O_8$  and KI with equal initial concentrations of the reactants
- 6. To determine solubility of sparingly soluble salts (anytwo) conductometrically.

## **Unit II: Inorganic Chemistry**

- 1. Identification of cations in a given mixture and Analytically separating them [From a mixture containing not more than two of the following: Pb(II), Ba(II), Ca(II), Sr (II), Cu(II), Cd(II), Mg(II), Zn(II), Fe(II), Fe(III), Ni(II), Co(II) Al(III), Cr(III)]
- 2. Crystallisation of potassium iodate and to estimate its purity before and after the separation.
- 3. Estimation of total hardness
- 4. Investigation of the raction between Copper supfate and Sodium Hydroxide (Standard EDTA solution to be provided to the learner).

## Unit III: Organic Chemistry

Short organic preparation and their purification: Use 0.5-1.0g of the organic compound.

Purify the product by recrystallization. Report theoretical yield, percentage yield and

melting point of the purified product.

## **Preparation of:**

- 1. Cyclohexanone oxime from cyclohexanone.
- 2. Glucosazone from dextrose or fructose
- 3. Tribromoaniline from aniline.
- 4. β-Naphthylbenzoate
- 5. m-Dinitrobenzene from nitrobenzene

- 6. Phthalic anhydride from phthalic acid by sublimation
- 7. Acetanilide from aniline
- 8. p-Bromoacetanilide from acetanilide
- 9. Iodoform from acetone
- (Any eight preparations)

# Semester IV Chemistry Practicals:

#### **Unit I: Physical Chemistry**

- 1. To determine standard EMF and the standard free energy change of Daniel cell potentiometrically .
- 2. To determine the amount of HCl in the given sample potentiometrically.
- 3. Compare the strengths of HCl and  $H_2SO_4$  by studying kinetics of acid hydrolysis of methyl acetate.
- 6. Industrial visit report.

#### **Unit II: Inorganic Chemistry**

- 1. Inorganic preparation Nickel dimethyl glyoxime using microscale method.
- 2. Complex cation *Tris* (ethylene diamine) nickel (II) thiosulphate.
- 3. Complex anion Sodium Hexanitrocobaltate (III) The aim of this experiment is to understand the preparation of a soluble cation (sodium) and a large anion hexanitrocobaltate(III) and its use to precipitate a large cation (potassium)
- 4. Inorganic salt Calcium or magnesium oxalate using PFHS technique

#### **Unit III: Organic Chemistry**

Qualitative Analysis of bi-functional organic compounds on the basis of

- 1. Prelimenary examination
- 2. Solubility profile
- 3. Detection of elements C, H, (O), N, S, X.
- 4. Detection of functional groups
- 5. Determination of physical constants (M.P/B.P)

Solid or liquid Compounds containing not more than two functional groups from among the following classes may be given for analysisto be given: Carboxylic acids, phenol, carbohydrates, aldehydes, ketones, ester, amides, nitro, anilides, amines, alkyl and aryl halides.

Students are expected to write balanced chemical reactions wherever necessary. (Minimum 6 compounds to be analyzed)

#### **Reference Books for Practicals:**

## Unit I:

- 1. Khosla B.D., Garg V.C. and Gulati A., Senior Practical Physical Chemistry, R. Chand and Co., New Delhi (2011).
- 2. Garland C. W., Nibler J.W. and Shoemaker D.P., Experiments in Physical Chemistry, 8th Ed., McGraw-Hill, New York (2003).
- 3. Halpern A.M. and McBane G.C., Experimental Physical Chemistry, 3rd Ed., W.H. Freeman and Co., New York (2003).
- 4. Athawale V.D. and Mathur P., Experimental Physical Chemistry, New Age International, New Delhi (2001)

## Unit II:

1. *Practical Inorganic Chemistry* by G. Marr and B. W. Rockett van Nostrand Reinhold Company (1972)

## Unit III:

- 1. Mann, F.G. & Saunders, B.C. Practical Organic Chemistry, Pearson Education (2009)
- Ahluwalia, V.K. & Aggarwal, R. Comprehensive Practical Organic Chemistry: Preparation and Quantitative Analysis, University Press (2000). Mann, F.G. & Saunders, B.C. Practical Organic Chemistry, Pearson Education (2009)
- Furniss, B.S.; Hannaford, A.J.; Smith, P.W.G.; Tatchell, A.R. Practical Organic Chemistry, 5<sup>th</sup> Ed., Pearson (2012)
- 4. Vogel, A.I., Tatchell, A.R., Furnis, B.S., Hannaford, A.J. & Smith, P.W.G., Textbook of Practical Organic Chemistry, Prentice-Hall, 5th edition, 1996

#### **Reference Books:**

### Unit I:

- 1. Barrow, G.M. Physical Chemistry Tata McGraw-Hill (2007).
- 2. Castellan, G.W. Physical Chemistry 4th Ed. Narosa (2004).
- 3. Kotz, J.C., Treichel, P.M. & Townsend, J.R. General Chemistry CengageLearning India Pvt.Ltd., New Delhi (2009).
- 4. Mahan, B.H. University Chemistry 3rd Ed. Narosa (1998).
- 5. Petrucci, R.H. General Chemistry 5th Ed. Macmillan Publishing Co., NewYork (1985).
- 6. K.L.Kapoor A textbook of Physical Chemistry3<sup>rd</sup> Ed. vol.1,2Macmillan Publishing Co., NewDelhi(2001)

## Unit II:

- 1. *Practical Inorganic Chemistry* by G. Marr and B. W. Rockett van Nostrand Reinhold Company (1972)
- 2. Inorganic Chemistry Gary Wulfsberg, Viva Book, First Indian Edition 2002
- 3. Quantitative Analysis R.A.Day, A.L. Underwood, sixth edition
- 4. Vogel's Textbook of quantitative chemical analysis J Mendham, R C Denny, J D Barnes, M Thomas, B Sivasankar

## 5. References.

- 6. Bruce H. Mahan, University Chemistry, Narosa publishing house pg. 611 to 683.
- 7. R. Gopalan, Universities Press India Pvt.Ltd. Inorganic Chemistry for Undergraduates.
- 8. Chemistry of Transition Elements Pg.- 608 679.
- 9. J. D. Lee, 4th Edn., Concise Inorganic Chemistry, ELBS, The group III elements Pg. 359- 648.
- 10. D. F. Shriver and P. W. Atkins, Inorganic chemistry, 3rd edition, Oxford University Press (1999) page 325-446.
- 11. Ramesh Kapoor and R.S. Chopra, Inorganic Chemistry, R. Chand publishers, New Delhi.
- 12. CNR Rao edited, University General Chemistry, 513-578.
- 13. James E. Huheey, Inorganic Chemistry: Principles of Structure and Reactivity,
- 14. Emeleus and Anderson, Modern Aspects of Inorganic Chemistry, page no. 435-463.
- 15. Cotton and Wilkinson, Advanced Inorganic Chemistry, 3<sup>rd</sup>. Edition.
- 16. Gary Wulfsberg, Inorganic chemistry, Viva Books Pvt,. Ltd. (2002).
- 17. Puri, Sharma and Kalia, Milestone publishers, Principles of Inorganic Chemistry, page 416-628.
- 18. Bruce H. Mahan, University Chemistry, Narosa publishing house.
- 19.R. Gopalan, Universities Press India Pvt.Ltd. Inorganic Chemistry for Undergraduates.
- 20.J. D. Lee, 4th Edn., Concise Inorganic Chemistry, ELBS
- 21.D. F. Shriver and P. W. Atkins, Inorganic chemistry, 3rd edition, Oxford University Press (1999)
- 22. Ramesh Kapoor and R.S. Chopra, Inorganic Chemistry, R. Chand publishers, New Delhi.
- 23.CNR Rao edited, University General Chemistry
- 24. James E. Huheey, Inorganic Chemistry: Principles of Structure and Reactivity,

25. Emeleus and Anderson, Modern Aspects of Inorganic Chemistry

- 26. Cotton and Wilkinson, Advanced Inorganic Chemistry, 3<sup>rd</sup>. Edition.
- 27. Gary Wulfsberg, Inorganic chemistry, Viva Books Pvt,. Ltd. (2002).
- 28. Puri, Sharma and Kalia, Milestone publishers, Principles of Inorganic Chemistry

#### Unit III:

- 1. Morrison, R. T. and Boyd, R. N. Organic Chemistry, Dorling Kindersley (India) Pvt. Ltd. (Pearson Education).2012
- 2. Finar, I. L. Organic Chemistry (Volume 1), Dorling Kindersley (India) Pvt. Ltd. (Pearson Education).
- 3. Finar, I. L. Organic Chemistry (Volume 2: Stereochemistry and the Chemistry of Natural Products), Dorling Kindersley (India) Pvt. Ltd. (Pearson Education)
- 4. Mc Murry, J.E. Fundamentals of Organic Chemistry, 7<sup>th</sup> Ed. Cengage Learning India Edition, 2013.
- 5. Clayden, J.; Greeves, N.; Warren, S.; Wothers, P.; Organic Chemistry, Oxford University Press.
- 6. Graham Solomons, T.W. Organic Chemistry, John Wiley & Sons, Inc.
- 7. Comprehensive Organic Chemistry- The synthesis and reactions of Organic Compounds, Derek barton ,W. David Ollis.
- 8. Kalsi, P. S. Textbook of Organic Chemistry 1<sup>st</sup> Ed., New Age International (P) Ltd. Pub.
- 9. Eliel, E. L. and Wilen, S. H. Stereochemistry of Organic Compounds, Wiley: London, 1994.
- 10. Kalsi, P. S. Stereochemistry Conformation and Mechanism, New Age International, 2005

# Semester III Paper III Basics in analytical Chemistry

## **Theory: 45 Lectures**

The Role of Analytical chemistry in various fields including non-chemistry fields such as Environmental Science, Pharmacy, Medicine, Life Sciences, Petrochemicals, Arts (like Painting) Forensic sciences and so on can never be underestimated. This course is expected to introduce the learner to this interesting field of Analytical Chemistry.

It is expected to provide the learner an overview of this very important branch of chemistry. After successful completion of this course the learner is expected to be familiar with the question of what is analysis, why it is required and the methods, techniques, procedures and protocols that may be used or required in the course of a given problem of analysis. The learner is also expected to appreciate the role of an Analytical Chemist and a Chemical Analyst.

Correctness or acceptability of the results of a given analysis and how to deal with wrong or erroneous results: when to reject them and when and how to retain them to be meaningful and/or acceptable are some other attributes expected as outcomes of learning this paper.

As such it is felt that this paper will be a subject of choice and interest for learners preferring a specialisation in Chemistry as well as to those who may have interests in other science fields as Physics, Botany, Zoology, Microbiology, Geochemistry and so on. **Goal:** 

To introduce the learner to an area of learning that is vital for the inherent nature of the subject itself but also is important and irreplaceable irrespective of the long term interest of specialisation or subject of interest of the learner.

## Unit I- Intorduction to Analytical Chemistry and Statistical Treatment of analytical data-I (15 L)

#### Scope/ Objectives:

Learners should be able to

- 1. Select a method of analysis
- 2. Decide how to identify a sample and prepare it for analysis
- 3. Select a procedure for analysis
- 4. Identify sources of possible errors in the results obtained.

(Problems including numericals expected wherever necessary)

#### 1.1. Role of Analytical Chemistry (9 L)

- 1.1.1. Language of analytical chemistry: important terms and their significance in Analytical Chemistry.
- 1.1.2. Purpose of Chemical Analysis; Analysis Based (i) On the nature of information required: (Proximate, Partial, Trace, Complete Analysis) and (ii) On the size of the sample used (Macro, semi-micro and micro analysis)
- 1.1.3. Classical and Non-Classical Methods of Analysis; their types and importance.

#### 1.2. Significance of Sampling in Analytical Chemistry

- 1.2.1. Terms involved in Sampling
- 1.2.2. Types of Sampling
- 1.2.3. Sampling techniques

#### **1.3.** Results of Analysis.

(6L)

- 1.3.1. Errors in Analysis and their types
- 1.3.2. Precision and Accuracy in Analysis
- 1.3.3. Corrections for Determinate Errors

(Problems including Numericals expected wherever required)

#### References:

- 1. Instrumental Analysis by Douglas A. Skoog, F. James Holler, Stanley R. Crouch
- 2. Instrumental methods of analysis by Willard, H.H.; Merritt, L.L. Jr.;
- Dean, J.A.; Settle,7<sup>th</sup> Edition
- 3. Fundamental of Analytical Chemistry by Douglas A. Skoog, West, F. James Holler,
- S. R. Crouch

4. Modern Analytical Chemistry by David Harvey, McGraw-Hill Higher Education

#### Unit II- Classical Methods of Analysis(15 L)

Objectives:

The main objectives of this unit is to

- > Introduce classical methods of chemical analysis.
- > Appreciate the various terms and types of titrimetric analysis.
- Ability to select proper titrimetric method
- > Appreciate the usefulness of the gravimetric method of analysis
- Identify a suitable gravimetric method
- Perform the required calculations involved in the analysis by titrimetry as well as gravimetry.

#### 2. Classical Methods of Analysis.

## **2.1.** Titrimetric Methods

- 2.1.1. Terms involved in Titrimetric methods of analysis. Comparing volumetry and Titrimetry
- 2.1.2. The Conditions suitable for titrimetry
- 2.1.3. Types of titrimetry Neutralisation (Acidimetry, alkalimetry), Redox,
   (Iodometry, Iodimetry,) Precipitation and Complexometric titrations and indicators used in these titrations
- 2.1.4. Tools of Titrimetry: Graduated glasswares and Callibration
- **2.2.** Standard solutions (Primary and Secondary standards in Titrimetry) and Calculations in Titrimetry.

#### **2.3.** Neutralisation Titrations

- 2.3.1. Concept of pH and its importance in Neutralisation Titrations
- 2.3.2. End point and Equivalence point of Neutralisation titrations
- 2.3.3. Determination of End point by using
  - i. Indicators causing colour change
  - ii. Change in potential, (by potentiometry)
  - iii. Change in conductance (by conductometry)
- 2.3.4. Construction of titration curve (on the basis of change in pH )of a titration of
  - i. Strong acid-weak base
  - ii. Strong base-weak acid

## 2.4. Gravimetric analysis (06 L)

- 2.4.1. General Introduction to Gravimetry.
- 2.4.2. Types of Gravimetric Methods -
- 2.4.3. Precipitation Gravimetry:
  - i. Steps involved in precipitation gravimetry analysis
  - ii. Conditions for precipitation
  - iii. Completion of precipitation,
  - iv. Role of Digestion, Filtration, Washing, Drying Ignition of precipitate.

#### (04L)

(04L)

v. Applications of Gravimetric Analysis: Determination of sulfur in organic compounds; Estimation of Nickel in Cu-Ni alloy using dimethyl glyoxime; Determination of Aluminum by converting it to its oxide.

[15 L]

## References:

1) Skoog et al. "Fundamentals of Analytical chemistry" Cengage Learning, Eight Edition, chapter 13, 14 and 15

- 2) Day and Underwood, "Quantitative analysis" prentice hall 1991, chapter3
- 3) S.M. Khopkar, "Basic Concepts of Analytical Chemistry", IInd Edition NewAge International Publisher
- 4) Gary D. Christan," Analytical Chemistry", VIth Edition, Wiley Students Edition, Chapter No 8,9,10
- 5) Fundamental of Analytical Chemistry by Douglas A. Skoog, West, F. James Holler, S. R. Crouch
- 6) Modern Analytical Chemistry, David Harvey (page numbers 232 -265)

#### Unit III: Instrumental Methods-I

Objectives:

On completing the learning of this unit the learner is expected to

- Know the various instrumental methods of analysis
- Advantages of using instruments to make measurements
- The various observable properties of a given analyte and the stimulus best suited for its analysis
- > Know about a generalized diagram of an analytical instrument
- > Select a suitable instrumental method for analysis
- > Appreciate the basic terms in spectrometry
- Use the relationship between absorbance (and its variations) and concentration of the analyte.
- > Chose a suitable method foe photometric titrations.

#### 3. Basic Concepts in Instrumental methods

- **3.1.** Relation between the Analyte, Stimulus and measurement of change in the observable property.
- 3.2. Block Diagram of an Analytical instrument.
- 3.3. Types of Analytical Instrumental methods based on
  - i. Optical interactions (eg. Spectrometry: uv-visible, Polarimetry)
  - ii. Electrochemical interactions (eg. Potentiometry, Conductometry,)

(03)

iii. Thermal interactions (eg. Thermogravimetry)

#### 3.4. Spectrometry

- 3.4.1. Interaction of electromagnetic radiation with matter: Absorption and Emission spectroscopy
- 3.4.2. Basic Terms: Radiant Power, Absorbance, Transmittance, Monochromatic

## (07 L)

light, Polychromatic light, Wavelength of maximum absorbance, Absorptivity and Molar Absorbtivity

- 3.4.3. Statement of Beer's Law and Lambert's Law, Combined Mathematical Expression of Beer -Lambert's Law, Validity of Beer-Lambert's Law, Deviations from Beer-Lambert's Law ((Real deviations, Instrumental deviations and Chemical deviations) (Numerical problems based on Beer-Lambert's Law)
- 3.4.4. Instrumentation for absorption spectroscopy: Colorimeters and Spectrophotometers
- 3.4.5. Block Diagrams for Single beam and Colorimeter, and Spectrophotometer (Principles, Construction and working-Details of Components expected i.e, source ,Sample holder , Filters/Monochromators, Detectors such as Photomultiplier tube)
- 3.4.6. Applications of UV-Visible Spectrophotometry (02 L)
  - (a) Qualitative analysis such as Identification of functional groups in Organic compounds ,Chromophores and Auxochrome,cis and trans isomers
  - (b) Quantitative analysis by Calibration curve method and
- 3.4.7. Photometric Titrations: Principle ,Instrumentation, Types of Photometric titration Curves with examples. (03L)

References:

1. Instrumental Methods of Chemical Analysis by Gurdeep R. Chatwal,

Sham K.Anand pp 2.107-2.148

- 2.Principles of Instrumental Analysis by Skoog, Holler, Nieman, 5<sup>th</sup> Edition pp 143-172.
- 3. Instrumental Methods of Analysis by Willard, Merritt, Dean, Settle

7<sup>th</sup> Edition pp 118-181.

# Semester III

# **Chemistry Practicals:**

## Paper III Basics in Analytical Chemistry

#### 1. Tools of Analytical Chemistry-I:

- a) Analytical glass wares like burettes, pipettes, Standard flasks, Separating funnels.
- b) Weighing tools such as two pan balance and monopan balance, digital balances:
- c) Incineration devices: Burners, Electrical Incinerators, Muffle Furnace,
- d) Drying Devices: Hot Air Oven, Microwave Oven, Descicators, Vacuum descicators
- e) Monochromators, Filters, Sample holders, Prisms, Diffraction Gratings, Photoemissive cells, Photomultiplier tubes

(The learner should draw diagrams and write-ups providing uses, care and maintenance of the items mentioned in (a) and principle, construction and uses of items (b) to (e) in his journal.

- 2. Gravimetric estimation of Nickel (II) as Ni-DMG and calculation of % error. (The learner is expected to know the role of the various reagents/chemicals used In the estimation, various steps involved. They should write the complete and Balanced chemical reaction for the formation of the Ni(DMG)<sub>2</sub> complex.
- 3. Colorimetric Determination of Copper Ions in given Solution by using calibration curve method and calculation of % error.

(The learner is expected to learn the relation between concentration and Absorbance, to draw a calibration curve, use the slope of the calibration curve and compare it with the calculated slope. They are also expected to state the error estimate of their results).

- 4. Determination of buffer capacity of acid buffer and basic buffer. (The learner is expected to learn the use pH meter, standardization of pH meter, use of Henderson's equation and calculation of buffer capacity)
- **5.** Estimation of Aspirin
- 6. Gravimetric estimation of barium ions using  $K_2CrO_4$  as precipitant calculation of % error.

(The learner is expected to learn the skills of using the counterpoise technique used in this gravimetric estimation; Using counterpoise method whatman No.42 for filtration. In such a case no incineration or use of silica crucible is required. They are also expected to state the error estimate of their results)

## Semester IV Paper III Basics in Analytical Chemistry -II **Theory: 45 Lectures** Unit –I -Methods of separation (15 L)

#### **Objectives**:

The learner is expected to understand

- The importance of separation in sample treatment  $\succ$
- $\triangleright$ Various methods of separations
- $\triangleright$ How to select a method of separation of an analyte from the matrix
- How a solute gets distributed between two immiscible phases
- AAA Principle of solvent extraction and various terms involved therein
- Effect of various parameters on solvent extraction of a solute
- $\triangleright$ Classification of Chromatographic methods
- $\triangleright$ Paper and thin layer chromatography and using them in practice.

# **1.** Separation Techniques in Analytical Chemistry

- **1.1.** An Introduction to Analytical Separations and its importance in analysis.
- 1.2. Estimation of an analyte without effecting separation.
- 1.3. Types of separation methods
- Based on Solubilities (Precipitation, Filtration Crystallisation) 1.3.1.
- Based on Gravity- Centrifugation 1.3.2.
- Based on volatility-Distillation; 1.3.3.

# (02 L)

- 1.3.4. Based on Electrical effects-Electrophoresis
- **1.3.5.** Based on retention capacity of a Stationary Phase -Chromatography;
- **1.3.6.** Based on distribution in two immiscible phases-Solvent Extraction;
- **1.3.7.** Based on capacity to exchange with a resin-Ion Exchange;
- **1.4. Electrophoresis:** Principles, Basic Instrumentation, Working and Application in separation of biomolecules like enzymes and DNA. (02L)

#### **1.5.** Solvent extraction

# (06 L)

- 1.5.1. Introduction, Nernst distribution Law, Distribution Ratio, Partition Coefficient.
- 1.5.2. Conditions of extraction: Equilibration time, Solvent volumes, temperature, pH.
- 1.5.3. Single step and multi step extraction, Percentage extraction for single step and multistep extraction. Separation factor.
- 1.5.4. Batch and continuous extraction
- 1.6. Chromatography :
- 1.6.1. Introduction to Chromatography
- 1.6.2. Classification of chromatographic methods based on stationary and mobile phase
- 1.6.3. Paper Chromatography: Principle, techniques and applications of Paper Chromatography in separation of cations.
- 1.6.4. Thin layer Chromatography Principle, technique and Applications in determining the purity of a given solute; Following progress of a given reaction .

#### References :

- 1. D.A. Skoog, D.M. West, F.J. Holler and CX.R. Crouch Fundamentals of Analytical chemistry, 8<sup>th</sup> edition
- 2. G.H. Morrison and H. Freiser, Solvent extraction in analytical chemistry
- 3. P. G. Swell and B. Clarke, Chromatographic separations, Analytical chemistry by open Learning, John Wiley and sons, 1987
- 4. Modern Analytical Chemistry, David Harvey (page numbers 596-606)
- 5. Modern Analytical Chemistry, David Harvey (page numbers 215 217)

#### Unit –II - Instrumental Methods-II

Objectives

On completing this unit the learner is

- Expected to appreciate the nature of interaction between applied electrical potential and the concentration of the analyte.
- > The nature of chemical reactions that influence potential of a given cell.
- ➤ Familiar with the various types of electrodes or half cells.
- > Appreciate the nature, need and importance of pH
- Expected to know the applications of the various instrumental methods dealt with in this unit.
- 2. Instruments based on the electrochemical properties of the analytes
- 2.1. Potentiometry:

(05 L)

- 2.1.1. Principle.
- 2.1.2. Role of Reference and indicator electrodes

## (05L)

(15 L)

- 2.1.3. Applications in Neutralisation reactions with reference to the titration of a Strong acid against a Strong Base (using quinhydrone electrode)
- 2.1.4. Graphical methods for detection of end points
- 2.2. pHmetry:

#### (04 L)

- 2.2.1. Principle
- 2.2.2. Types of pH meters.
- 2.2.3. Principle, Construction Working and Care of Combined Glass electrode
- 2.2.4. Applications in Titrimetry (Strtong acid-Strong Base) biological and environmental analysis.
- 2.3. Conductometry:

#### (06 L)

- 2.3.1. Principle
- 2.3.2. Conductivity cell its construction and care
- 2.3.3. Applications in Neutralisation Titrimetry with respect to
  - i. Strong Acid-Strong Base
  - ii. Strong Acid-Weak Base
  - iii. Strong Base-weak Acid
  - iv. Weak Acid- Weak Base.
- 2.3.4. Advantages & limitations of conductometric titrations.

#### References:

- Principles of Instrumental analysis, D. A. Skoog, 3<sup>rd</sup> edition, Saunders college publishing. Chapters: 20, 23 Page nos: 600 - 605, 631, 704 - 711.
- 2) Vogel's Text book of quantitative inorganic analysis, 4<sup>th</sup> edition,
   ELBS/ Longman. Chapters: XIV, XV
   Page nos: 566 601, 615 625.
- 3) Instrumental methods of analysis, B. K. Sharma, Goel publishing house.Miscellaneous methods: Chapters: 1, 3, 4 Page nos: 1 14, 21 57.

# Unit III- Statistical Treatment of analytical data --II (15 L)

## Objectives:

On completing this unit the learner is expected to understand

- i) The use of statistical methods in chemical analysis.
- ii) The nature of indeterminate errors
- iii) The randomness of such errors and its distribution around a correct or acceptable result
- iv) Computation of Confidence limits and confidence interval
- v) Test for rejection of doubtful result
- vi) Method to draw best fitting straight line

#### 3.1.Nature of Indeterminate Errors: (03L)

- 3.1.1. The true and acceptable value of a result of analysis
- 3.1.2. Measures of central tendency: mean, median. mode, average
- 3.1.3. Measures of dispersion: Absolute deviation, relative deviation, relative average deviation, standard deviation,(s,sigma) variance, coefficient of variation

#### 3.2.Distribution of random errors: (02L)

- 3.2.1. Gaussian distribution curve.
- 3.2.2. Equation and salient features of Gaussian distribution curve

# 3.3.Concept of Confidence limits and confidence interval and its computation using

- (03 L)
  - (i) Population standard deviation
  - (ii) Student's *t* test
  - (iii) Range

## **3.4.**Criteria for rejection of doubtful result (02 L)

- (i) 2.5 d rule
- (ii) 4.0 d rule
- (iii) Q test

## **3.5.Test of Significance**

- (i) Null hypothesis
- (ii) F-test (variance ratio test)

## 3.6. Graphical representation of data and obtaining best fitting straight line (03 L)

(02 L)

- (a) For line passing through origin
- (b) For line not passing through origin
- [ Numerical problems wherever possible, expected ]

## References:

- 1. Modern Analytical Chemistry, David Harvey (page numbers 53-84)
- 2. Fundamentals of analytical chemistry Skoog and West

# Semester IV Chemistry Practicals: Paper III Elective ( Basics in analytical Chemistry )

- 1. Tools of Analytical Chemistry-II
- a. Filtration Flasks, Funnels, Separating Funnels, Distillation apparatus, Vacuum Distillation assembly, Centrifuge machine, Electrophoresis apparatus.
- b. Development chamber for chromatography
- c. Electrodes like Reference Electrodes and Indicator Electrodes (with respect to care and maintenance.)
- d. Conductivity cell (with respect to care and maintenance.)
- e. Combined Glass electrode (with respect to care and maintenance.)
- f. Types of Salt Bridges and preparation of any one or use of salt bridge, its effect on the potential of a given electrode/cell

(The learner should draw diagrams and write-ups providing uses of the items mentioned in (a and b) and Principle, Construction care and Uses of items (c) to (f) in his journal.)

2. Paper chromatography: Separation of cations like Fe(III), Ni(II) and Cu(II) in a sample.

3. Separation of a solute between two immiscible solvents to determine the distribution ratio and/or extraction efficiency. (Solutes could be as their aqueous solutions and the organic solvent ethyl acetate) Suggested solute for the distribution study: Fe (III) in aqueous solutions.

(The learner is expected to learn the technique of solvent extraction by using separating funnel, method to estimate the concentrations of the solute distributed in the two immiscible phases, determination of the extraction efficiency)

- 4. Conductometric titration: Estimation of given acid by conductometric titration with strong base and calculation of % error. (The learner is expected to learn the handling of the conductometer and the conductivity cell, determination of end point by plotting a graph. They are also expected to state the error estimate of their results).
- 5. Estimation of Fe(II) in the given solution by titrating against  $K_2Cr_2O_7$  potentiometrically and calculation of % error. (The learner is expected to learn the handling of the potentiometer, use of Platinum electrode and reference electrode like SCE. They will learn to determine end point by plotting a graph. They are also expected to state the error estimate of their results).
- 6. Gravimetric estimation of Sulfate as BaSO<sub>4</sub> and calculation of % error. (The learner is expected to write a balanced chemical reaction, need for digestion of the precipitate and the skill required to carry out the incineration and to estimate the % error.) (The learner is expected to write a balanced chemical reaction, need for digestion of the precipitate and the skill required to carry out the incineration and to estimate the % error.)

#### **REFERENCES:**

For paper III

- 1. D. A. Skoog, D. M. West, F. J. Holler, and S. R. Crouch, Analytical Chemistry: An Introduction, 7th ed., Chapter 15, pp. 345-381.
- 2. A.I. Vogel. "Textbook of Quantitative Inorganic Analysis," Longman, London (1961).
- 3. R.V. Dilts. "Analytical Chemistry. Methods of Separation," van Nostrand, N.Y. (1974).
- 4. Some Experiments for B. Tech in Chemistry & Chemical Technology compiled by Prof. J.B.BARUAH, Mrs. Abhilasha Mohan Baruah and Mr. Parikshit Gogoi