

SWAMI RAMANAND TEERTH MARATHWADA UNIVERSITY,
NANDED

CHOICE BASED CREDIT SYSTEM (CBCS)

SEMESTER PATTERN
Post Graduate (PG) Programme in Chemistry
(Affiliated Colleges)
w. e. f. Academic year 2015-16

SYLLABUS
M. Sc. SECOND YEAR [III & IV semester]
ANALYTICAL CHEMISTRY

Board of Studies in Chemistry
Swami Ramanand Teerth Marathwada University, Nanded

SWAMI RAMANAND TEERTH MARATHWADA UNIVERSITY, NANDED
Choice Based Credit System (CBCS)
Draft Syllabus Prescribed for
M. Sc. Second Year, ANALYTICAL CHEMISTRY
Semester-III & IV

4. Analytical Chemistry (Semester III & IV)

Semester	Paper	Course No.	Course	Periods / week	Total Periods	Credit	
Theory III	XV	CH-531	Advanced Spectroscopic Methods	04	60	04	
	XVI	CH-532/4	Fundamentals of Analytical Chemistry	04	60	04	
	XVII	CH-533/4	Chromatography in Chemical analysis -I	04	60	04	
	XVIII	CH-534/4	Instrumental methods of chemical analysis – I	04	60	04	
	XIX		Seminar			01	
						Total	17
Theory IV	XX	CH-541/4	Industrial Analytical Chemistry	04	60	04	
	XXI	CH-542/4	Analytical methods	04	60	04	
	XXII	CH-543/4	Chromatography and other Process in Chemical analysis-II	04	60	04	
	XXIII	CH-544/4	Instrumental methods of chemical analysis - II	04	60	04	
	XXIV		Seminar			01	
						Total	17
Practical III & IV	XXV	CH-501/4	Laboratory Course-V	06	144	04	
	XXVI	CH-502/4	Laboratory Course-VI	06	144	04	
	XXVII	CH-503/4	Laboratory Course-VII	06	144	04	
	XXVIII	CH-504/4	Laboratory Course-VIII	06	144	04	
						Total	16
						Grant Total	50 Credits

Instructions

- I] Each Laboratory Course of 6 Hrs duration should be completed in 6 Hrs per day.
- II] Assessment shall consist of continuous assessment (CA) and end of Semester Exam. (ESE).
- III] 75% for ESE and 25% for CA.
- IV] Evaluation of Seminar should be from departmental panel of experts.
- V] Visit to Sophisticated Instrumentation Centre / Industrial tour.

Draft Syllabus Prescribed for
M. Sc. Second Year, ANALYTICAL CHEMISTRY
Semester-III & IV

4. ANALYTICAL CHEMISTRY (SEMESTER III & IV)

Semester	Paper	Course No.	External (ESE)	Internal (CA)	Total Credits (Marks)
Theory III	XV	CH-531	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XVI	CH-532/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XVII	CH-533/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XVIII	CH-534/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XIX		25 Marks		01
					17
Theory IV	XX	CH-541/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XXI	CH-542/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XXII	CH-543/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XXIII	CH-544/4	75 Marks	2 Tests: 15 marks + Assignment: 10 Marks = 25 marks	04 (100)
	XXIV		25 Marks		01
					17
Practical III & IV	XXV	CH-501/4	75 Marks	2 Tests: 25 marks	04 (100)
	XXVI	CH-502/4	75 Marks	2 Tests: 25 marks	04 (100)
	XXVII	CH-503/4	75 Marks	2 Tests: 25 marks	04 (100)
	XXVIII	CH-504/4	75 Marks	2 Tests: 25 marks	04 (100)
					16
Total Credits Sem III & IV + Lab Course = 17 + 17 + 16 = 50					

**M. Sc. Second Year, Semester-III Paper–XV,
[CH-531]
Advanced Spectroscopic Methods**

Credits: 04

Periods: 60

SM-1: UV-Vis Spectroscopy:
SM-2: IR spectroscopy:
SM-3: NMR Spectroscopy (Organic):
SM-4: NMR Spectroscopy (Inorganic):
SM-5: Mass Spectroscopy:
SM-6: Moissabaur Spectroscopy:
SM-7: Structural problems:

SM-1: UV-Vis Spectroscopy:

06P

Fieser-Woodward rules for conjugated dienes and carbonyl compounds, Fieser-Kuhn rules for polyenes. UV spectra of aromatic compounds and heteroaromatic compounds. Calculation of λ_{\max} for the benzene derivatives (R-C₆H₄-Co-G) by A. I. Scott empirical rules.

SM-2: IR spectroscopy:

10P

a) Organic IR Spectroscopy: Recapitulation, Characteristic vibration frequencies of alkanes, alkenes, alkynes, aromatic compounds, alcohols, ethers, phenols and amines. Detailed study of vibrational frequencies of carbonyl compounds Ketones, aldehydes, esters, amides, acids, anhydride, Lactose, lactams and conjugated carbonyl compounds. Factors affecting group frequencies: overtones, combination bands and Fermi-resonance. FITR and sampling technique.

b) Inorganic IR Spectroscopy: Structural information from vibrational spectra: Group frequencies, Characteristic band stretching frequencies, Mode of vibrations of linear and non-linear molecules, deformation, frequencies of carbonyl metal complexes, pattern of group frequencies, mode of bonding of ambidentate ligands, Cyanides, Ethylenediamine and Diketone complexes.

SM-3: NMR Spectroscopy (Organic):

12P

- a) ¹H NMR:** General introduction and definitions, Chemical shift, Spin-spin interaction, shielding mechanism of measurement of chemical shift values and correlation for protons bonded to carbon (aliphatic, olefinic, aldehyde and aromatic) and other nuclei (alcohols, phenols, enols, carboxylic acids, amines, amides and mercapto). Factors affecting chemical shift. Deuterium exchange. Spin-spin coupling, factors affecting coupling constant. Complex spin-spin interaction between two and three nuclei. Simplification of complex spectra, nuclear magnetic double resonance, contact shift reagents, solvent effects. Fourier transform technique. Nuclear Over-Hauser effect (NOE). Resonance of other nuclei; ¹⁹F and ³¹P.
- b) ¹³C NMR:** Resolution and multiplicity of ¹³C NMR, 1H-decoupling, noise decoupling, broad band decoupling; Deuterium, fluorine and phosphorus coupling; NOE signal enhancement, off-resonance, proton decoupling, Structural applications of CMR. DEPT; Introduction to 2D-NMR: COSY, NOESY, DEPT, INPET, APT, INADEQUATE.

SM-4: NMR Spectroscopy (Inorganic): **08P**

- a) Basic principle of NMR spectroscopy and applications to Paramagnetic compounds and metal nuclei of Pt ¹⁹⁵ and Sn ¹¹⁹.
- b) Basic principle and applications of ESR spectroscopy to different free radical molecules and transition metal ion complexes.

SM-5: Mass Spectroscopy: **06P**

Theory, instrumentation and modifications; Unit mass and molecular ions; Important terms- singly and doubly charged ions, metastable peak, base peak, isotopic mass peaks, relative intensity, FTMS, etc.; Recognition of M⁺ ion peak; General fragmentation rules: Fragmentation of various classes of organic molecules, including compounds containing oxygen, sulphur, nitrogen and halogens; α -, β -, allylic and benzylic cleavage; McLafferty rearrangement.

SM-6: Moissabaur Spectroscopy: **08P**

Basic principle of Moissabaur Spectroscopy, applications on the basis of isomer shifts, electric quadrupole interactions. Elucidation of structure of I₂Br₂Cl₄, I₂Cl₆, Fe⁺² and Fe⁺³ complexes and Sn⁺² and Sn⁺⁴ compounds

SM-7: Structural problems: **10P**

- a) Combined problems on UV, IR, NMR and Mass spectral data for structure determination.
- b) Elucidations of structure of organic molecules using spectra (IR & NMR).

Reference Books:

1. Spectroscopic identification of Organic Compounds, R. M. Silverstern, G. C. Bassler and T. C. Morrill.
2. Introduction to NMR spectroscopy, R. J. Abraham, J. Fisher and P. Loftus.
3. Application of spectroscopy of organic compounds – J. R. Dyer.
4. Spectroscopy of organic compounds, P. S. Kalsi.
5. Organic Spectroscopy, William Kamp.
6. Organic Chemistry, R. T. Morrison and R. N. Boyd.
7. Practical NMR spectroscopy, M. L. Martin, J. J. Delpenck and G. J. Martin.
8. Spectroscopic methods in organic Chemistry, D. H. William, I. Fleming.
9. Fundamentals of Molecular spectroscopy – C.N.Banwel

M. Sc. Second Year, Semester-III
Paper–XVI, [CH-532/4]
Fundamentals of Analytical Chemistry
(FAC)

Credits: 04

Periods: 60

FAC-1: Introduction
FAC-2: Errors and Evaluation of Analytical Data:
FAC-3: Mole concept and Chemical calculations
FAC-4: General concepts of Chemical Equilibrium:

FAC-1.Introduction:

11P

Introduction, classification of analytical methods, classical and instrumental types of instrumental analysis, selection of analytical method, Laboratory operations and practices. Analytical balance. Techniques of weighing, weighting errors. Volumetric glassware – cleaning, calibration of glassware, Sampling,. Sampling statistics., sampling of solids, Liquids and gases. Dissolution and decomposition of samples, Laboratory note books, safety in Laboratory. Problems.

FAC-2.Errors and Evaluation of Analytical Data:

16P

Definition of terms, Precision, deviation, mean deviation, standard deviation, accuracy, absolute errors, types of errors determinate, indeterminate and gross, sources of errors and their effect on final result, Methods of reporting analytical data., statistical evaluation of data. Indeterminate errors, Linear least squares methods, correlation coefficient. Significant figures, computation rules, Use of scientific calculators. Problems.

FAC-3.Mole concept and Chemical calculations:

17P

Mole, concentration units, solubility, molarity, molality, formality , percentage concentration, dilution, equivalence, milliequivalents, millimoles, ppm, ppb, titer, calculations of equivalents in acid base, redox, precipitation, complexation, reactions. Calculation of concentration from density and percentage, Problems.

FAC-4.General concepts of Chemical Equilibrium:

16P

Chemical reaction, rate constant, Gibb's free energy and equilibrium constant. LeChatelier's principle,. Factors affecting chemical equilibrium constant-temperature, pressure, concentration, catalyst, Completeness of reactions, equilibrium constants for dissociating species, calculations using equilibrium, constants, Common ion effect, activity and activity co-efficient, diverse ion effect, thermodynamic equilibrium constant. Problems.

Books Suggested:

1. L.Erdey, gravimetric analysis. Oxford: Pergamon, 1965
2. J.s.Fritz, Acid-Base Titration in Non-aqueous Solvents, Boston : Allyn and Bacon, 1973
3. W.F.Hillebrand, G.E.F. Lundell, H.A.Bright and J.I.Hoffman, applied Inorganioc Analysis 2nd ed. New York: Wikky, 1953
4. I.M.Kolthoff, V.a.Stenger and R.Bekher, Volumetric Analysis, New York: Inter Science 1942-57, Three volumes.
5. R.Bock, Decomposition Methods in analytical Chemistry, New York, Wiley 1979
6. R.A.Day, Jr.A.L.Underwood,, Quantitative Analysis, Sixth ediction 1991, Preutict Hall, INC, Engle Wood Clits, N.J.U.S.A.
7. J.H.Kennedy, Analytical Chemistry: Principles, 2nd ed. Saunders College Publishing, New York, 1990
8. D.A.Skoog, D.M.West and F.J.Holler. Fundamentals of Analytical Chemistry. 6th ed.Saunders College Publishing, New York 1992
9. F.W.Fifield and D.Kealey. Principles and Practice of Analytical Chemistry. 3rd ed. Blackie Glasgopw and Lonmdon (1990)
10. J.H.Jeffery et.al., Vogel's Text book of Quantitative Chemical Analysis, 5th ed. ELND Longman (1989)

M. Sc. Second Year, Semester-III
Paper–XVII, [CH-533/4]
Chromatography in Chemical Analysis I
(CCA-I)

Credits: 04

Periods: 60

CCA-1 General principles of chromatography

CCA-2 Paper Chromatography

CCA-3 Thin layer Chromatography

CCA-4 Column chromatography

CCA-5 Ion Exchange chromatography

CCA-6 Gas-liquid chromatography

CCA 1. General principles of chromatography: 17P

Definition of terms, migration rates of solutes, partition ratio, retention time, dead time, relation between retention time and partition ratio, rate of solute migration (The capacity factor), differential migration rates (The selectivity factor), efficiency of chromatographic columns, rate theory of chromatography, column efficiency, plate height, number of theoretical plates, definition of plate height, experimental evaluation of H & N. Kinetic variables affecting band broadening, theory of band broadening, optimization of column performance, column resolution, optimization techniques. Summary of important relationships for chromatography, applications of chromatography qualitative and quantitative. Problems.

CCA 2. Paper Chromatography 08P

Sample solution, application of sample solution to the paper, choice of solvent, development of chromatogram, drying of the paper, location of spots on paper chromatogram. Limits of detection on chromatograms, concept of R values, applications.

CCA 3. Thin layer Chromatography 08P

Preparation of chromatoplates, chromatoshets and ready-prepared chromatoplates, choice of medium, selection of solvent, development of chromatogram. Location of substances on chromatogram, applications.

CCA 4. Column chromatography 08P

Preparation of column. Types of adsorbents. Selection of solvents and eluents. Column resolution. Applications.

CCA 5. Ion Exchange chromatography 07P

Ion exchangers, ion exchange resins, action of ion exchange equilibria, selectivity coefficient, separation factor, ion exchange capacity, column operation, changing the ionic forms, strongly acidic cation exchangers, weakly acidic cation exchangers, strongly basic anion exchangers, weakly basic anion exchangers, applications. Problems

CCA 6. Gas-liquid chromatography 12P

Principles of GLC, instruments for GLC, carrier gas supply, sample injection system, column, column thermostating, detectors, liquid phases for GLC, applications of GLC – qualitative and quantitative. Problems

Books Suggested

1. B.L.Karger, L.R.Snyder and C.Horvath. An Introduction to Separation Science, New York: Wiley 1973
2. J.M.Miller, Separation Methods in Chemical Analysis, New York: Wiley 1975
3. W.Rieman and H.F. Walton, Ion Exchange in Analytical Chemistry. Oxford Pergamon 1970
4. W.J.Williams, Handbook of anion Determination. London. Butter Worthy, 1979
5. Synder and Kirkland, Introduction to Modern Liquid Chromatography, 2nd ed. (1973)
6. R.b.Fisher and D.G.Peters, Quantitative Chemical Analysis, 3rd 1968
7. G.D.Christian, Analytical Chemistry, 4th Ed. John Wiley and sons. New York, 1986.

M. Sc. Second Year, Semester-III
Paper–XVIII, [CH-534/4]
Instrumental Methods of Chemical Analysis – I
(IMCA-I)

Credits: 04

Periods: 60

IMCA-1 Introduction to Absorption and Emission Spectroscopy

IMCA-2 Flame Emission and Atomic Absorption Spectrophotometry

IMCA-3 Optical methods

IMCA-4 Thermal analysis:

IMCA-1. Introduction to Absorption and Emission Spectroscopy: 08P

Nature of electromagnetic radiation, electromagnetic spectrum, atomic energy levels, molecular electronic levels, vibrational energy levels, absorption spectra, emission spectra. Problems

IMCA-2. Flame Emission and Atomic Absorption Spectrophotometry: 19P

Atomization, sample atomization, continuous atomizers, discrete atomizers, types and sources of atomic spectra, atomic emission spectra, atomic absorption spectra, atomic fluorescence spectra, atomic line widths, line broadening from uncertainty effects. Doppler shift, pressure broadening effect of temperature on atomic spectra, interference by molecular spectra during atomization. Flame atomization types of flames, Flame structure, flame atomizers, electrothermal atomizers. Atomic absorption, radiation sources. Single beam and double beam spectrophotometers. Spectral interference, chemical interferences, analytical techniques, in AAS, application of AAS. Flame photometry instrumentation, interferences, analytical techniques, application of flame photometry. Problems.

IMCA- 3 Optical methods: 19P

A. Refractometry: Refractive index n_{air} , n_{vac} , conversion of n_{air} to n_{vac} , principle of working of Abbe's refractometer, immersion refractometer, Pulfrich refractometer, Hilger-Chance, recording refractometers, application of Abbe's refractometer. Problems.

B. Polarimetry: Theory, optical rotatory dispersion and circular dichroism theory, measurement of optical rotation, Faraday effect, multicomponent analysis, calculations of polarimetry and saccharimetry, applications of optical rotatory, dispersion and circular dichroism, polarimeter, automatic recording spectro polarimeters, applications. Problems.

C. Nephelometry and Turbidimetry: Theory, standards (NTU), instrumentation, applications.

IMCA-4. Thermal analysis: 14P

Differential, thermal analysis and differential scanning calorimetry, thermogravimetry, methodology of DSC (or DTA) and TG, thermo mechanical analysis, thermometric titrations and applications. Problems.

Books Suggested:

1. F.W.Fifield and D.K.Kealey, Principles and Practice of Analytical Chemistry, 3rd ed. Blackie Ghsgow and London (1990)
2. D.P.Shoemaker, C.W.Garland, and J.W.Nibler, Expeiments in Physical Chemistry, 5th ed. McGrtaw Hill international Ediution (1989)
3. H.H.Willard, L.L.Merritt, J.A.Dean and F.a.Settle, Instrumental Methods of Analysis.Wadsworth Publishing Company U.S.A.
4. D.A.Skoog, J.J.Leary, Principles of Instrumental analysis. Salanrdes college publishing foundation Edn. New York.
5. E,J.Bair. Introduction to Chemical Instrumentation. Mc Graw-Hill, New York, 1962. 6. E.D.Olsen, Modern Optical Methods of Analysis, Mc.Graw Hill, New York, 1975.
7. J.A.Dean and T.C.Rains, Eds., Flame Emission and Atomic Absorption spectrometry: thoery Vol I, 1969, components and Techniques Vol II, 1971: Elements and Matrics Vol III 1975, Marcel-Dekkar, New York.
8. Kolthoff,I.M. and P.J.Elving, Eds., Trease on analytical Chemistry, Chap, 11-14 and 16, Vol I Part I Wiley – Interscience, New York, 1984.
9. F.J.Welcher, Ed., standard Methods of Chemical Analysis, 6th ed., Vol.3 Part A. Van Nostrand Rein hold. New York, 1966.

M. Sc. Second Year, Semester-IV
Paper-XX, [CH-541/4]
Industrial Analytical Chemistry
(IAC)

Credits: 04

Periods: 60

IAC-1 Analysis of Oils and fats

IAC-2 Introduction to Pharmacopeia

IAC-3 Analysis of Plastics and Synthetic fibers

IAC-4 Process and Analysis of sugar and fermentation industries

IAC-5 Analysis of Fuels

IAC-6 Rubber Analysis

IAC-7 Analysis of Glass

IAC-8 Analysis of Paints and Pigments

IAC-9 Analysis of Cement

IAC1. Analysis of Oils and fats

07P

Formulae of fatty acids, composition of some common oils and fats, composition of some drying oils, composition of peanut oil, sesame oil, castor oil, jute seed oil, butter fat, animal fat. Classification of oil, drying oils, semi-drying oils, non drying oils, waxes Tests of oils, fats and waxes, saponification value, acid value, Iodine value uses of oils and fats.

IAC2 Introduction to Pharmacopeia

06P

Introduction, different assay of drug: Loss on drying, storage, assay by chromatographic method and spectral technique.

IAC3 Analysis of Plastics and Synthetic fibers

06P

Plastics: Definitions, classification of Plastics, thermo plastics, properties and uses Synthetic fibers
Classification of synthetic fibers, Rayon or Artificial Silk properties and uses

IAC4 Process and Analysis of sugar and fermentation industries

08P

Sugar Industries Manufacture of White crystalline sugar Two steps method, extraction of Juice, classification of Juice ,evaporation of juice to make syrup, treatment of molasses, testing and estimation of sugar Fermentation Industries Introduction, Enzymes, yeast, brewery yeast, distillery yeast, wine yeast, preparation of culture east on a large scale, pure culture of distillery yeast, Manufacture of Vinegar , Uses of Vinegar.

IAC5 Analysis of Fuels

07P

Introduction, color and consistency, origin, composition, classification, distillation, Matural gasoline, Aviation Gasoline, Cracking process, Octane number, cetaen number, Antiknocking compounds.

IAC6 Rubber Analysis

07P

Natural Rubber, coagulation of latex, chemical and physical properties of rubber, properties of synthetic rubber and uses.

IAC7 Analysis of Glass

07P

Physical properties of glass, Classification of glass, Chemical properties of glass, effect of different constituent in glass, chemical reactions, Types of glass.

IAC8 Analysis of Paints and Pigments

06P

Types of paints, smoke point, flash point, characteristics of a good paint, paint failure, zinc white manufacture, properties and uses, manufacturing of red lead.

IAC9 Analysis of Cement

06P

Introduction, Composition of cement, Analysis of cement: Analysis of Silica, Calcium etc by gravimetric process and volumetric process.

Books Suggested

1. Analysis and characterization of oils, fats and fat products. Vol. 2 H. A. Boekenooen
2. Lipid Analysis in Oils and Fats R.J. Hamilton
3. Analysis of Oils and Fats 1st Edition by R. J. Hamilton J. B. Rossell
4. Martindale: The Complete Drug Reference, 38th Edition
5. Martindale: The Complete Drug Reference on Medicines Complete
6. van Tellingen "Pliny's pharmacopoeia or the Roman treat". Netherlands heart journal 15 (3): 118–
7. Philip K. Hitti (cf. Kasem Ajram (1992), Miracle of Islamic Science
8. Kohan, Melvin (1995). Nylon Plastics Handbook. Munich: Carl Hanser Verlag
9. J. Robinson (ed) "The Oxford Companion to Wine" Third Edition pg 267-269
10. H. Johnson Vintage: The Story of Wine pg 16 Simon and Schuster 1989 ISBN 0-671-68702-
11. Guide to Combustion Analysis and Fuel Efficiency Paperback – August 1, 2007 by Erik Rasmussen
12. .Medium and High Efficiency Gas Furnaces Paperback Richard Jazwin
13. .Analysis of Rubber and Rubber-like Polymers, : Loadman, M.J.
14. . Rubber Analysis - Polymers, Compounds and Products Paperback –by M. J. Forrest
15. .Application of Differential Thermal Analysis in Cement Chemistry by V.S. Ramachandran
16. Cement Chemistry and Physics for Civil Engineers by Wolfgang Czernin
17. Textbook of analytical chemistry by Y. R. Sharma
18. A textbook of analytical chemistry by Alka Gupta.
19. Analytical chemistry by H. Kaur
20. Textbook of industrial chemistry & metallurgical analysis by S. K. Jain.
21. University practical chemistry by Solamn
22. J. D. Christian, Analytical chemistry , 4th edition John Wiley and sons. New York , 1986.
23. Quantitative and qualitative analysis by Vogel.
24. Analytical chemistry by S. M. Khopkar
25. Fundamentals of analytical chemistry by Skoog and West.
26. Principles of analytical chemistry by J. H. Kennedy.
27. Quantities analysis by R. A. Day And A. L. Underwood.

M. Sc. Second Year, Semester-IV
Paper–XXI, [CH-542/4]
Analytical Methods
(AM)

Credits: 04

Periods: 60

AM-1 Classical Methods of Chemical analysis

AM-2 Analysis of water and Soil:

AM-3 Analysis of body Fluids and Drugs

AM-4 Analysis of Food

AM 1. Classical Methods of Chemical analysis: 18P

A). Definition of terms. Primary and secondary standards used in neutralization, redox precipitations and complexations titrations, theory of acid-base, redox, precipitation. Complexometric titrations, indicators in titrimetric analysis, theory of indicators, non-aqueous titrations.

B). Gravimetric analysis: Unit operations in gravimetric analysis, preparation of sample solution, precipitation, types of precipitates, filtration,. Washing of precipitation, drying, ignition, weighing, and gravimetric factor. Use of organic precipitants. Problems

AM 2. Analysis of water and Soil: 14P

A) Analysis of water:

Water pollution, water pollutant and their sources, determination of physicochemical parameters, acidity, alkalinity, hardness, chloride, sulphate, nitrate, fluoride, nitrogen in various forms, heavy metals, Pb, Hg, Cd, As, etc, organic matter water pollution laws and standards.

B) Analysis of soil

Moisture, Ph, total nitrogen, phosphorous, silica, lime, magnesia, manganese, sulphur, and alkali salts.

AM 3. Analysis of body Fluids and Drugs: 14P

A) Clinical Chemistry:

Composition of blood, collection and preservation of samples, clinical analysis, serum electrolytes, blood glucose, blood urea nitrogen, uric acid, albumin, globulin, barbiturates, acid and alkaline phosphates.

B) Drug Analysis:

Classification of drugs, narcotics and dangerous drugs, screening by gas and thin layer chromatography and Spectrophotometric measurement.

AM 4. Analysis of Food: 14P

Moisture, ash, crude protein, fat, crude fiber, carbohydrates, Ca, K, Na and PO_4^{-3} .

Food adulteration- Common adulteration in food, contamination of food stuff.

Microscopic examination of foods for adulterants. Pesticide analysis in food products.

Extraction and purification of sample.

Books Suggested:

1. L.Erdey, gravimetric analysis. Oxford: Pergamon, 1965
2. J.s.Fritz, Acid-Base Titration in Non-aqueous Solvents, Boston : Allyn and Bacon, 1973
3. W.F.Hillebrand, G.E.F. Lundell, H.A.Bright and J.I.Hoffman, applied Inorganico Analysis 2nd ed. NewYork:
4. I.M.Kolthoff, V.a.Stenger and R.Bekher, Volumetric Analysis, New York: Inter Science 1942-57,
5. R.Bock, Decomposition Methods in analytical Chemistry, New York, Wiley 1979
6. R.A.Day, Jr.A.L.Underwood., Quantitative Analysis, Sixth ediction 1991, Preutict Hall, INC, Engle Wood
7. J.H.Kennedy, Analytical Chemistry: Principles, 2nd ed. Saunders College Publishing, New York, 1990
8. D.A.Skoog, D.M.West and F.J.Holler. fundamentals of Analytical Chemistry. 6th ed.Saunders College Publishing, New York 1992
9. F.W.Fifield and D.Kealey. Principles and Practice of Analytical Chemistry. 3rd ed. Blackie Glasgopw and Lonmdon (1990)
10. J.H.Jeffery et.al., Vogel's Text book of Quantitative Chemical Analysis, 5th ed. ELND Longman (1989)

M. Sc. Second Year, Semester-IV
Paper–XXII, [CH-543/4]
Chromatography and Other Process in Chemical Analysis-II
(CCA-II)

Credits: 04

Periods: 60

CCA-1 High performance liquid chromatography

CCA-2 Liquid-Liquid Extraction

CCA-3 Process Instruments and Automatic Analysis

CCA 1. High performance liquid chromatography: 20P

Instruments for HPLC, mobile phase reservoirs and solvent treatment systems, pumping systems, sample injection systems, columns for HPLC, detectors, high performance partition chromatography, banded phase packing, normal and reverse phase packing, choice of mobile and stationary phases, applications – high performance adsorption chromatography, stationary and mobile phases, applications, HPTLC . Problems

CCA 2. Liquid-Liquid Extraction: 20P

Nernst's distribution law, distribution coefficient, distribution ratio, factors affecting K_d and d , efficiency of extraction, selectivity of extraction, extraction systems, multiple extractions, types of extraction procedure – simple, exhaustive extraction, counter current extractions.

Applications : Extractive separation of metal ions as chelates, effect of P^H and reagent concentration ion distribution ratios, extractions with diphenylthiocarbazone, extraction with 8-hydroxyquinoline, the extraction of metal chlorides, extraction of nitrates, crown ethers and their applications.

CCA 3. Process Instruments and Automatic Analysis: 20P

Introduction, industrial process analyzers, method based on bulk properties, infrared process analyzers,, oxygen analyzers, on-line potentiometric analyzers, continuous on-line process control, automatic chemical analyzers, automatic elemental analyzers applications.

Books Suggested

1. B.L.Karger, L.R.Snyder and C.Horvath. An Introduction to Separation Science, New York: Wiley 1973
2. J.M.Miller, Separation Methods in Chemical Analysis, New York: Wiley 1975
3. W.Rieman and H.F. Walton, Ion Exchange in Analytical Chemistry. Oxford Pergamon 1970
4. W.J.Williams, Handbook of anion Determination. London. Butter Worths, 1979
5. Snyder and Kirkland, Introduction to Modern Liquid Chromatography, 2nd ed.
6. R.b.Fisher and D.G.Peters, Quantitative Chemical Analysis, 3rd 1968
7. Morrison, G.H. and H.Frieser : Solvent Extraction in analytical Chemistry. New Yoprk: John Wileyand sons. Inc.1957.
8. De, A.K., S.M.Khopkar and R.A.Chalmers. Solvent Extraction of Metals. New York : VanNostrand Reinholdeo, 1970
9. Sekine, T and Y.Gasegawa. Solvent Extraction Chemistry, New York; Marcel Dekker, Inc, 1977

M. Sc. Second Year, Semester-IV
Paper–XXIII, [CH-544/4]
Instrumental Methods Of Chemical Analysis – II
(IMCA-II)

Credits: 04

Periods: 60

IMCA-1 UV & Visible Spectrophotometry

IMCA-2 Infra red Spectrophotometry

IMCA-3 Mass Spectrophotometry

IMCA-4. Electroanalytical Methods

IMCA-5 Radio analytical Chemistry

IMCA 1. UV & Visible Spectrophotometry: 13P

Fundamental laws, Spectrophotometry accuracy, photometric precision, quantitative methodology, instrumentation – radiation sources, filters, monochromators, grating monochromator systems, detectors, read out modules, instruments for UV and visible Spectrophotometry, correlation of electronic absorption spectra with molecular structure, applications of UV and visible spectrophotometry for single, multicomponent analysis, complex study, photometric titrations. Problems.

IMCA 2. Infra red Spectrophotometry: 09P

Theory, correlation of infra red spectra with molecular structure, instrumentation – radiation sources, detectors, thermal detectors, photon detectors, spectrophotometers, sample handling, gases, liquids solutions, films, mulls, pallette technique, cell thickness, multiple internal reflectance, quantitative analysis. Problems.

IMCA 3. Mass Spectrophotometry: 10P

Main components of mass spectrometers, sample inlet system, ion sources, ion collection system (Ion donor), mass analyses, vacuum system and pumping system. Electrostatic accelerating system, resolution in mass spectrometer, mass spectrometers, interfacing chromatography and mass spectrometry (GC/MS), GC/MS) instruments, quantitative analysis of mixtures, correlation of mass spectra with molecular structure.

IMCA 4. Electroanalytical Methods 15P

A) Potentiometry: Electrochemical cells, electrode potentials, reference electrodes, glass-membrane electrodes, liquid membrane electrodes, gas sensing electrodes, electrometric measurements of P^H and PI, potentiometric titrations, indicator electrodes, location of the equivalence point, types of potentiometric titrations, acid-base titrations in non-aqueous solvents, applications, Problems.

B) Paleography: Theoretical principles, residual current, migration current, diffusion current, half wave potential, quantitative techniques, wave height, internal standard method, method of standard addition, measurement of wave height, polarographs, applications Problems.

C) Conductometry: Electrolytic conductivity, conductance, conductivity, equivalent conductance, measurement of electrolytic conductance, conductance cells, conductometric titrations, types of conductometric titrations, neutralization, precipitation and complexometric, applications. Problems.

IMCA 5. Radio analytical Chemistry**13P**

Detection and measurement of radioactive radiation. Elementary working and principles of GM and scintillations counter and gamma ray spectrometer, neutron radiation sources ,neutron activation analysis –applications. Method of preparation of some commonly used radioactive isotope and their uses in varies field's .Isotope dilution analysis and its application

Books Suggested:

1. F.W.Fifield and D.K.Kealey, Principles and Practice of Analytical Chemistry, 3rd ed. Blackie Ghsgow and London (1990)
2. D.P.Shoemaker, C.W.Garland, and J.W.Nibler, Expeiments in Physical Chemistry, 5th ed. McGrtaw Hill international Ediution (1989)
3. H.H.Willard, L.L.Merritt, J.A.Dean and F.a.Settle, Instrumental Methods of Analysis.Wadsworth Publishing Company U.S.A.
4. D.A.Skoog, J.J.Leary, Principles of Instrumental analysis. Salanrdes college publishing foundation Edn. New York.
5. E,J.Bair. Introduction to Chemical Instrumentation. Mc Graw-Hill, New York, 1962.
6. Middleditch, B.S.Ed., Practical Mass Spectrometry, Plenum, New York 1979
7. J.J.Lingane Electronanalytical Chemistry, Iind ed. Wiley – Inter science, New York, 1958
8. Kolthoff,I.M. and P.J.Elving, Eds., Trease on analytical Chemistry, Chap, 11-14 and 16, Vol I Part I Wiley – Interscience, New York, 1984.
9. F.J.Welcher, Ed., standard Methods of Chemical Analysis, 6th ed., Vol.3 Part A. Van Nostrand Rein hold. New York, 1966.

**M. Sc. Second Year
Laboratory Course-V,
Paper– XXV, CH-501**

Credits: 04

Periods: 144

- 1 To determine the amount of vitamin c in the given tablet using statistical method of analysis
- 2 To separate Fe and Mg by solvent extraction method
- 3 Analysis of Brass Alloy
- 4 Analysis of Dolomite ore
- 5 To determine the acidity of given urine sample titrimetrically
- 6 To determine the amount of ascorbic acid from the given sample of Lemon juice
- 7 To determine the rancidity of oil
- 8 To estimate the amount of cholesterol in blood sample by calorimetrically
- 9 To determine assay of aspirin tablet
- 10 To estimate amount of sulphanilamide group in given sample of sulpha drug using std AgNO₃
- 11 To estimate the amount of glucose in Honey
- 12 Analysis of Wheat flour
- 13 Determine the specific gravity of an oil sample
- 14 Analysis of egg sample
- 15 To estimate the amount of Tannin in tea sample
- 16 To estimate the amount of Caffeine in coffee sample
- 17 To estimate the amount of Casein in milk sample
- 18 To estimate the amount of Ca and Mg in face powder
- 19 Analysis of Portland cement
- 20 Analysis of Paints
- 21 Assay of Amoxicilin Tablet

**M. Sc. Second Year
Laboratory Course-VI,
Paper– XXVI, CH-502**

Credits: 04

Periods: 144

1. General

1. Calibrations of volumetric apparatus. Burette, pipettes, volumetric flask
2. Calibration of mercury thermometers.

2. Titrimetric analysis

1. Preparation of standard potassium hydrogen phthalate solution and standardization of barium hydroxide solution
2. Preparation of constant boiling point HCl and standardization of NaOH solution.
3. Determination of replaceable hydrogen in given unknown acid
4. Determination of HCO_3^- (Bicarbonate) in blood using back titration
5. Determination of total nitrogen content of fertilizer.
6. Determination of iron in iron pills
7. Determination of “volume strength” of hydrogen peroxide

3. Gravimetric analysis

- 1 Determination of aluminium in alum (turti)
- 2 Determination of magnesium in a given sample of milk of magnesia.
- 3 Determination of sulphur content of a coal sample
- 4 Determination of thiamine hydrochloride as thiamine silicotungstate

4. Paper Chromatography

1. Separation of metal ions by paper chromatography
2. Separation of indicators by paper chromatography

5. Thin layer chromatography

1. Separation of dye stuffs on activated silica gel
2. Separation of amino acids on deactivated silica gel

6. Ion exchange chromatography (any two)

1. Determination of ion exchange capacity of cation / anion exchanger
2. Separation and determination of zinc and magnesium in a given mixture solution
3. Separation and determination of nickel and cobalt in a given mixture solution
4. Determination of sodium with the aid of cation exchanger
5. Determination of fluoride with the aid of cation exchanger
6. Determination of total cation concentration in water

7. Gas chromatography

1. Construction of simple apparatus for gas chromatography and separation of chloromethane
2. Determination of aluminium and chromium as their acetyl acetonates

- 8. Spectrophotometry (visible) (any two)**
1. Determination of Mn in steel / soil
 2. Determination of fluoride in water by zirconil alizarin red method
 3. Determination of Ti and V in a mixture solution (simultaneous determination)
 4. Determination of stability constant of the colored complex by Jobs method / mole ratio method/ slope ratio method
- 9. Flame Photometry**
1. Determination of calcium in blood serum / calcium bills
 2. Determination of Na / K / Li/ Ca in sample solution
- 10. Refractometry (any two)**
1. Determination of refractive index, specific refractivity, molar refractivity of given liquid at a given temperature
 2. Determination of molecular refractivity of solid substance
 3. Determination of composition of an unknown mixture of two liquids
 4. Determination of atomic refractivity of C, H and O from methyl acetate, ethyl acetate and n-hexane.
- 11. Polorimetry (any two)**
1. Determination of percentage of D-glucose, in given sample
 2. Determination of two optically active compounds in the given mixture solution
 3. Determination of specific, molecular and intrinsic rotation of an optically active compound.
 4. Determination of percentage purity of d-tartaric acid / cane sugar

**M. Sc. Second Year
Laboratory Course-VII,
Paper– XXVII CH-503**

Credits: 04

Periods: 144

(Any one from each section)

1. LFT

1. Indicate the splitting of the d-levels and the number of electrons in each level for each of the following complexes.

- a) $\text{Ni}(\text{NH}_3)_6^{+2}$ (Paramagnetic)
- b) $\text{Trans-Ni}(\text{CH}_3)_4\text{Cl}_2$ (Paramagnetic, tetragonal, Δ for $\text{NH}_3 > \text{Cl}^-$)
- c) $\text{Cu}(\text{NH}_3)_4^{+2}$
- d) $\text{Trans-Ni}(\text{CN})_4(\text{H}_2\text{O})_2^{-2}$
- e) NiCl_4^{-2} (tetrahedral)
- f) $\text{Mn}(\text{acac})_3$ (acac = acetylacetonate)
- g) $\text{Cr}(\text{CN})_6^{-3}$
- h) FeO_4^{-2} (tetrahedral)
- i) CuCl_4^{-2} (tetrahedral)
- j) $\text{Co}(\text{NH}_3)_6^{+2}$

2. Of the complexes in a, e, f, g, h, i and j above, which would you expect to be distorted?

3. Compare the CFSE in $\text{Co}(\text{NH}_3)_6^{+2}$ and $\text{Co}(\text{NH}_3)_6^{+3}$

4. a) Calculate the relative energies as a function of Dq and P for both the high spin (HS) and low spin (LS) octahedral and HS tetrahedral aquo complexes of $\text{Co}(\text{II})$ and $\text{Fe}(\text{II})$. On the basis of these calculations state which configuration is the most energetically favorable for each of these ions.

b) On the basis of the CFSE's alone, predict which ion should show the greater tendency form tetrahedral complexes.

c) Is this prediction upheld by the calculations made above and if not, why?

2. Applications Of Infrared And Raman Spectroscopy (Any One)

The spectrum of $\text{Co}(\text{NH}_3)_6(\text{ClO}_4)_3$ has absorption bands at 3320 cm^{-1} , 3240 cm^{-1} , 1630 cm^{-1} , 1352 cm^{-1} , and 803 cm^{-1} . For purposes of assignment the molecule can be Treated as a C_{3v} molecule. Use the V_n symbolism to label the bands and also describe them as bends, stretches etc.

3. Magnetic Chemistry

1. a. $\text{Co}(\text{N}_2\text{H}_4)_2\text{Cl}_2$ has a magnetic moment of 3.9 BM. Is hydrazine bidentate? Propose a structure.

b. How could electronic spectroscopy be employed to support the conclusion in a.

2. In which of the following tetrahedral complexes would you expect contributions from spin orbit coupling? V^{+3} , Cr^{+3} , Cu^{+3} , Co^{+2} , Fe^{+2} , Mn^{+2}

3. In which of the following spin-paired square planar complexes would you expect contributions? d^2 , d^3 , d^4 , d^5 , d^6
 4. Why is $\text{Fe}_2(\text{CO})_9$ with three bridging and six terminal carbonyl is diamagnetic?
 5. Explain why mixing of a D_{4h} component in with T_d ground state lowers the Moment nickel (II) complexes.
 6. What is expected magnetic moment for Er^{+3} ?
4. **Mossbauer Spectroscopy** Problems 7.1 to 7.6.
 5. **Vibrational Spectroscopy**
Problems 5.9 to 5.15
 6. **Potentiometry (any Two)**
 1. Determination of strong acid in a given solution,. Titrating with strong base using hydrogen electrode
 2. Determination of standard electrode potential of quinhydrone electrode
 3. Determination of formal redox potential of $\text{Fe}^{2+}/\text{Fe}^{3+}$, $\text{Ce}^{3+}/\text{Ce}^{4+}$ systems at Different concentration of strong acid
 4. Determination of ionic product of water.
 7. **Conductometry (any Two)**
 1. Determination of λ_o of HCl , NaCl , CH_3COONa hence find out λ_o of CH_3COOH by Kohlraush law
 2. Determination of degree of hydrolysis and hydrolysis constant of aniline hydrochloride / sodium acetate / ammonium chloride
 3. Determination of titration error of acid-base titration
 4. Determination of dissociation constant of benzoic acid / lactic acid / n-propionic acid
 8. **Solvent extraction (any four)**
 1. Determination of distribution coefficient of iodine between carbon tetra chloride and water.
 2. Determination of K_d of succinic acid between diethyl ether and water.
 3. To study the distribution of benzoic acid between benzene and water
 4. Determination of formula of cuprammonium complex
 5. Determination of percentage of fatty acids in soft soaps
 6. Determination of morphine in a given sample of morphine sulphate tablets.
 7. Determination of iron, in iron and aluminium ion mixture solution, by chloride extraction
 8. Determination of lead by dithiozone method
 9. Determination beryllium as acetyl acetone complex
 10. Determination of silver by extractions as its ion association complex with 1:10 phenolphtholine and bromo pyrogallol red.
 11. Determination of nickel by synergistic extraction.
 12. To study effect of contraction of hydrochloric acid on extraction of iron by chloride extraction.

9. Non-aqueous titrations (any Two)

1. Preparation of acetic perchloric acid and its standardization
2. Determination of percentage purity of amine using standard acetic perchloric acid solution
3. Determination of ephedrine hydrochloride in given drug sample.

Books Suggested:

2. Structural methods in Inorganic Chemistry by E.A.V. Ebsworth.
3. Physico chemical methods in Inorganic Chemistry by Drago

**M. Sc. Second Year
Laboratory Course-VIII,
Paper– XXVIII, CH-504(Project + Seminar)**

Credits: 04 (Project: 75 + Seminar 25)

Periods: 144

The students will develop utilities such as analytical spectra, simulation programmes that will supplement laboratory exercises in their subject of specialization. For this, variety of small research project designed by the teacher based on the interest of the student and capabilities should be worked out.

The students will have to give at least one seminar in each semester in their subject of specialization. For this, submission of synopsis of seminar delivered by every student is compulsory which is to be produced before examiner of practical examination. The project work carried out during the year should be presented in power point presentation in presence of University Examiners.

Important Notes:

1. In house laboratory training of a week / fortnight in industry / research institute is compulsory.
2. Visit to the National research Institutes for example NCL, NPL, I I C T, BARC,SIC etc is compulsory
- 3 Educational tour is compulsory
- 4 Aims and objectives of all the experiments are well defined.

By

Dr. Subhash M. Lonkar

MEMBER,

Board of Studies in Chemistry

And

Chairman in M. Sc. II year Analytical Chemistry.

Swami Ramanand Teerth Marathawada University, Nanded

Dr. Dashrat R.Munde

MEMBER,

Board of Studies in Chemistry
S.R.T.M.U,Nanded.

Dr. Shivaji B.Patwari

CHAIRMAN,

Board of Studies in Chemistry
S.R.T.M.U,Nanded.

CHOICE BASED CREDIT SYSTEM (CBCS)
SEMESTER PATTERN
Post Graduate (PG) Programme in Chemistry
(Affiliated Colleges)
w. e. f. Academic year 2015-16
Question paper Model
(For all papers)

Mark: 75

Time: 3 hrs

Q 1. Solve any three out of five.

Marks – 15

- (a)
- (b)
- (c)
- (d)
- (e)

Q 2. Attempt any three out of five.

Marks – 15

- (a)
- (b)
- (c)
- (d)
- (e)

Q 3. Solve. (A)

OR (A)

Marks – 08

(B)

OR (B)

Marks – 07

Q 4. Solve. (A)

OR (A)

Marks – 08

(B)

OR (B)

Marks – 07

Q 5. (A) Select the correct alternative from the following –

Marks -05

(B) Write short notes on any two –

Marks - 10

- (a)
- (b)
- (c)