

M. Sc. - II Analytical Chemistry

Semester – III

CHA-5301: Analytical Method Development & Validation, Nanotechnology (48 L+ 12 T) (4 Credit)

Section I- Analytical Method Development & Validation (24 L+ 06 T) (2 Credit)

1. Assay Validation and Inter Laboratory Transfer

Introduction, fundamental definitions, Essential principles of method transfer, method validation report, the inter-laboratory qualification (ILQ) process, (4 L, Ref 1 pp. 3 to 14)

2. Statistical Analysis and analytical Figure of Merit

Introduction, Errors (gross errors, systematic errors, random errors), accuracy, how to reduce systematic errors Validation parameters: Accuracy, precision, mean and standard deviation, calibration, (Linear response functions (linear regression-errors in slope and the intercept, error in the estimate of concentration, standard additions), non-linear response functions and Weighted regression analysis, internal standards), selectivity and specificity (Chromatographic methods), limits of detections (spectrophotometric methods, Chromatographic methods and related techniques, receptor binding assay), limit of Quantification, sensitivity, ruggedness and robustness, analyte stability in the sample matrix, mean and standard deviation, reliability of results, confidence interval, comparison of results, comparison of two means of two samples (14 L, Ref-1 pp 15 to 68, Ref- 2 pp 145-197)

3. Overview of World Wide Regulations

(2 L, Ref-1, pp 75 to 98)

4. Specific methods and Applications: Dissolution Studies

Introduction, Dissolution test, Apparatus – USP type –I and II, Sampling and analytical instrumentation, Single point test Vs. Dissolution profile, Calibration, regulatory guidelines, analytical validation, linearity, accuracy, precision, specificity.

(4 L, Ref-1, pp 75 to 98 and 169 to 182)

References:

1. Development and validation of Analytical Methods, Progress Pharmaceutical and Biomedical Analysis, Vol-3, Edited by Chitofer M. Riley and Tomas W. Rosanske (Elvier)
2. Vogel's Textbook of quantitative Chemical Analysis, sixth Ed., Mendham, Denney, Barnes, Thomas, Pub: Pearson Education.
3. Handbook of modern pharmaceutical analysis, edited by SatinderAhuja and Stephen Scypinski, Academic Press, Separation science Series, Vol-3
4. HPLC method Development for pharmaceuticals, Edited by SatinderAhuja and Henrik Rasmussen, Academic Press, Separation science Series, Vol-8
5. Practical HPLC method Development, Snyder, Kirkiand, Glajch, Wiley India Pvt. Ltd.

Section II- Nanotechnology (24 L+ 06 T) (2 Credit)

1. Nanotechnology and carbon allotropes

Introduction, carbon allotropes- fullerene (C-60, C-80, C-240) ,nanostructures, properties (mechanical, optical, electrical), applications, Nano cutting tool, sensors for aerospace and defence, temperature and smoke sensor (6 L)

2. Carbon nanostructure

Introduction, nanotubes and its types, carbon molecules, carbon clusters, types of carbon nanotubes, properties of carbon nanotubes, preparation of nanotubes (methodologies), Applications of carbon nanotubes. (10 L)

3. The basic tools of nanotechnology

Nanostructure identification by using X-ray diffraction methods (XRD), Scanning Electron Microscope (SEM), Scanning Probe Microscope (SPM), Scanning Transmission Electron Microscopy (STEM), Atomic Force Microscopy (AFM) (6 L)

4. Biomedical applications

Introduction, biological science, photodynamic therapy in targeted drugs, biomedical sensor and biosensor, quantum dot technology in cancer treatment, nanoparticles as drug carrier (2 L)

References:

1. Introduction to nanotechnology , C.P. Poole, Jr. & F.J. Owens, John Wiley & sons (2009)
2. Nano biotechnology, Subbiah Balagi, MJP publishers, India(2010)
3. The Chemistry of nanomaterials Volume,1 C.N.Rao, A Muller & A.K.Cheetham,
4. Nano, the essentials, T.Pradeep, Tata McGraw Hill new Delhi,2007
5. Nanostructures & nanomaterial's -Synthesis-properties and applications, G. Cao, Imperical college press, London 2004

CHA-5302: Electrochemical Methods and Food Analysis
(48 L+ 12 T) (4 Credit)

Section I- Electrochemical Methods of Analysis (24 L+ 06 T) (2 Credit)

1 Coulometry

Current voltage relationship during an electrolysis, Operating cell an at fixed applied potential, Electrolysis at constant working electrode potential, Coulometric methods of analysis, Faradays laws of electrolysis, Instrumentations-Constant current and constant voltage instruments, potentiostatic coulometry-Instrumentation and applications, coulometric titrations (Amperostatic coulometry)-Apparatus and applications, advantages and limitations, problems. (6 L)

2 Voltammetry and polarographic methods of analysis (14 L)

A) Polarography (linear scan polarography):

Polarographic principles, Instrumentation (different types of microelectrode such as dropping mercury electrode, the static drop mercury electrode, rotating disc and ring disc electrode, cell for polarography, reference and counter electrode and circuit diagram), polarogram and polarographic currents, charging or capacitive current, role of supporting electrolyte, factors affecting on polarographic wave, Ilkovic Equation, advantages and disadvantages of DME, polarographic maxima and maxima suppressors, interference due to dissolved oxygen, Applications (qualitative analysis, quantitative analysis by calibration curve and standard addition methods), specific examples of analysis – analysis of Cu, Cd, Zn, Pb, etc. from tap water and alloys., problems.

B) Hydrodynamic Voltametry and its applications:

Volatametric detectors in chromatography, flow injection analysis, Volatametric oxygen sensors, amperometric titration).

C) Pulse Polarography:

Different types of excitation signals in pulse polarography, Differential pulse polarography, square wave polarography, Stripping method. Voltametry Withultra microelectrode, Applications of these technique Cu and Zn from tap water by differential pulse polarography and by square wave polarography, Vitamin-C by differential pulse polarography, Determination of Pb in tap water by stripping method.

D) Cyclic Voltametry:

Principle of cyclic Voltammetry, cyclic voltamogram of $K_3[Fe(CN)_6]$ and parathion, criteria of reversibility of electrochemical reactions, quasireversible and irreversible processes.

4 Amperometry:

Principle, Instrumentation, typical applications, amperometric titrations, chrono-amperometry and chrono-potentiometry. (4 L)

Section II- Food analysis (24 L+ 06 T) (2 Credit)

1. Carbohydrates:

Definition, classification, and functions, Analysis of carbohydrates from food sample by different method i) volumetric determination by Fehling's solution, ii) Colorimetric analysis of carbohydrates by Folin Wu method, Nelson Somyogi method, iii) total carbohydrates by Anthrone method, iv) Estimation of starch by anthrone method, v) Determination of amylase, vi) Estimation of pectic substances (gravimetric and colorimetric method), vii) Estimation of crude fibers (5 L, Ref 9 and 10)

2. Proteins:

Definitions and functions, Analysis of proteins by Kjeldahl's method, analysis of protein by Lowry method, Estimation of amino acids by colorimetric method, Estimation of food grain for methionine content, Protein digestibility in vitro, Protein efficiency and net protein ratio, Determination of net protein utilization, digestibility and biological value, Polyacrylamide gel electrophoresis of proteins **(5 L, Ref-1 and 2)**

3. Analysis of Lipids:

Estimation of oil in oilseeds, Estimation of free fatty acids, Saponification value of oils, iodine value, Determination of acid value of oil, determination of peroxide value of oil, Identification and quantification of fatty acids **(4 L, Ref 1 and 2)**

4. Determination of food preservatives:

Definition, SO₂ legislation and determination by Tanners method, Nitrate and nitrites legislation and determination, boric acid legislation and determination, Benzoic acid legislation and determination, 4-hydroxybenzoate legislation and determination, ascorbic acid legislation and determination. Sweeteners: Saccharine identification and determination, Colours: Identification by general methods, Natural colours. **(6 L, Ref 2)**

5. Milk:

Analysis of milk and milk products: Composition of milk, analysis of milk with respect to pH, acidity, fates, casein content, lactose content, mineral content, adulteration of milk. **(2 L, Ref 1 and 2)**

References:

- 1) Biochemical Methods, S Sadashivan, A.Manickam; New Age Publication, 3rd Edn
- 2) Introduction to instrumental analysis, R. D. Broun, Mc Graw Hill (1987)
- 3) Instrumental methods of chemical analysis, H. Willard, L.Merrit, J.A. Dean and F.A. Settle. Sixth edition CBS (1986)
- 4) Fundamentals of analytical chemistry, D. A. Skoog, D. M. West and H. J. Holler sixth edition (1992)
- 5) Principles of Instrumental Analysis, Skoog, West, Niemann.
- 6) Vogel Text Book of quantitative analysis 6th Ed.
- 7) J. chemical education, 60,302 to 308 (1983)
- 8) Cyclic Voltammetry and frontiers of electrochemistry, N.Noel and K.I. Vasu IBH, New Delhi (1990)

CHA-5303 Pharmaceutical Analysis

(48 L+ 12 T) (4 Credit)

Section I- Tests, Assay and Roll of FDA (24 L+ 06 T) (2 Credit)

1. A) Apparatus for test and assay, cleaning of glassware

B) Role of FDA in Pharmaceutical Industries:

Definitions of Drug & Cosmetics, Substandard Drugs, Role of FDA, Introduction to New Drug, Development of New Drugs- Selection of Area,, Phase I, Phase II, Phase III Applications to FDA for formulation and marketing of new drug. Stability studies and Shelf life fixation. (6 L)

2. Biological Tests & Assay:

Introduction to biological assay, Biological assay of Heparin sodium, Determination of Amylase activity, Determination of Photolytic Activity, Test for Insulin in solution, Biological Assay of Tetanus Antitoxin, Test for Undue Toxicity. (5 L)

3. Microbiological Tests and Assays:

Microbiological test for Antibiotics. Standard preparation and units of activity, Test organisms and Inoculums, Cylinder-plate assay receptacles, Turbidimetric assay receptacles, Assay Designs, Cylinder plate or Cup-plate method, Two level fractional assay, Test for Sterility, (7 L)

4. Physical Test, Determinations, Limit tests and Sterilization:

A) Disintegration Test for Tablets and Capsules, B) Dissolution Test for Tablets and Capsules, C) moisture / water content by Karl-Fischer titration, limit tests for arsenic, heavy metals, iron, lead, sulphate, chloride, D) Ash, sulphated ash, E) Methods for Sterilization Steam Sterilization, Dry heat sterilization, Sterilization by Filtration, Gas Sterilization, Sterilization by Ionizing radiation, Sterilization by heating with Bactericides, Water for Pharmaceutical use. (6 L)

Section II- Analysis and quality control (24 L+ 06 T) (2 Credit)

1. Analysis of vegetable Drugs:

Vegetable drugs: Sampling, foreign organic matter, ash value, acid soluble ash, acid insoluble ash, sulphated ash, Extraction of alkaloids. (4 L)

2. Sources of Impurities in Pharmaceutical raw materials & finished products, Shelf life of pharmaceutical product:

Raw materials, Method of manufacture, Atmospheric contaminations, Cross contamination, Microbial contamination, Container contamination, Packaging errors, Chemical instability, Temperature effect and Physical changes, shelf life of pharmaceutical product and determination of shelf life. (4 L)

3. Standardization and quality control of different raw materials and dosage form:

Analysis of raw materials with respect to identification, other or related substances, loss on drying, and Assay as per IP, i) adrenaline, ii) Niacin amide iii) Cephalexin, iv) ferrous fumarate, v) isoniazid and vi) paracetamol. Problems based on assay of these materials. Brief introduction to different dosage forms with the IP requirements Analytical methods for the following- Tablets, different types of tablets, uniformity in weight (aspirin) additives used in tablet manufacture, capsules, types of capsules, (Rifampicin) Powders (Sodium benzoate),

Solutions (saline NaCl) Suspensions (barium sulphate –limit test for impurity) Mouthwashes, (Ointments (salicylic acid) and creams Dimethicone by IR) Injections (Mannitol), ophthalmic preparations (sulphacteamine), Aerosols (salbutamol), Blood products and 6 reporting protocols. . Problems based on assay of these materials. **(16 L)**

References:

- 1) Indian Pharmacopeia, Volume I and II.
- 2) Practical Pharmaceutical chemistry, A.H.Beckett & J.B.Stenlake, third edition, volume 1.
- 3) Remington's Pharmaceutical sciences.
- 4) Ansel's Pharmaceutical Analysis

CHA-5304 Analytical Spectroscopy

(48 L+ 12 T) (4 Credit)

Section I- Spectroscopic techniques (24 L+ 06 T) (2 Credit)

1 Electron spectroscopy:

Introduction, principle of electron spectroscopy for chemical analysis(FSCA). Satellite peaks, spectral splitting, chemical shifts in FSCA. Apparatus used for ESCA, X-ray source, samples, Analysers, Detectors, Chemical analysis using ESCA, Applications, Auger electron microscopy and Ultraviolet photoelectron spectroscopy. **(6 L, Ref 1 and 6)**

2 X- ray Methods of Analysis:

Principle, Theory- X-ray spectral lines, X-ray tube, X-ray emission, Absorptive Apparatus: Sources, Collimation, sample handling, wavelength dispersive devices, Energy dispersive devices, detectors, readout device, Chemical analysis using X-ray absorption, X-ray Fluorescence- instrumentation and chemical analysis, X-ray Diffraction, Chemical analysis with X-ray diffraction, numerical problems. **(10 L, Ref 1 and 6)**

3 An Introduction to Microscopy (surface characterization techniques):

Limitations of the Human Eye, the X-ray Microscope, the Transmission Electron Microscope, the Scanning Electron Microscope, Scanning Transmission Electron Microscope, Analytical Electron Microscopy, Scanning-Probe Microscopes, the transmission electron microscope **(8 L, Ref 8 and 6)**

Section II- Luminescence and NMR Study (24 L+ 06 T) (2 Credit)

1. Chemiluminescence:

Introduction, principle, types. Measurement of chemiluminescence, Instrumentation, quantitative chemiluminescence, Gas phase Chemiluminescence analysis, Chemiluminescent titrations, electro-chemiluminescence. **(6 L, Ref 1)**

2. Fluorescence and phosphorescence:

Introduction, Fluorescence, photo luminescent theory, electron transitions during photoluminescence, factors affecting photoluminescence, luminescent apparatus, optical extractive sources, wavelength selectors, detectors ad readout devices, photo luminescent spectra, photo luminescent analysis, analysis of non-photoluminating compounds, determinations of mixtures, specific examples of analysis using photoluminescence, problems **(6 L, Ref 1 and 7)**

3. Nuclear magnetic resonance spectroscopy:

¹H-NMR: Introduction, theory, Instrumentation, Chemical shifts, spin-spin splitting, protons on heteroatom's, coupling protons with other nuclei, solvents, qualitative and quantitative analysis, problems.

¹³C NMR: Introduction, interpretation ¹³C NMR spectra, Chemical shifts, Spin coupling, quantitative analysis, problems.

2-D NMR: introduction, ¹H - ¹H connectivity, ¹H - ¹³C connectivity, ¹³C - ¹³C connectivity, Through space ¹H - ¹H proximity, option and how to use them, problems.

(12 L, Ref 1, 5 and 6)

References:

- 1) Introduction to instrumental analysis, R.D. Braun, MC. Graw Hill-Interl. edn.
- 2) Analytical spectroscopy, Kamlesh Bansal- First edition.
- 3) Instrumental methods of chemical analysis, Willard, Dean and Merittee- Sixth edition.
- 4) Analytical chemistry principles, John H Kenedey- 2nd edn, Saunders college publ.
- 5) Spectroscopic identification of organic compounds Silverstrine, Bassler, Morrill, 5th edn. John Wiley and sons.
- 6) Analytical chemistry, Ed by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed., Wiley- VCH.
- 7) Vogel's Textbook of quantitative Chemical Analysis,., Mendham, Denney, Barnes, Thomas, Sixth Ed ,Pub: Pearson Education.
- 8) Electron microscopy in the study of material, P.J Grundy and G.A.Jones, Edward Arnold.

Practical course I

CHA-5305: Analysis of Materials (4 Credit)

- 1 Analysis of ilmenite ore
- 2 Analysis of Dolomite ore for Ca, Mg and silicate material
- 3 Analysis of bauxite ore
- 4 Determination of aluminum and magnesium from magnesium alloy
- 5 Analysis of Bronze with respect to copper and tin
- 6 Analysis of nichrome alloy with respect to nickel and chromium
- 7 Analysis of cement with respect to SiO_2 , Calcium, iron, Magnesium and aluminum
- 8 Determination of nitrogen, phosphorous and potassium from fertilizer
- 9 Determination of iron detergent sample
- 10 Determination of organic carbon from soil sample
- 11 Determination of COD from waste water sample
- 12 Determination of magnesium from talcum powder
- 13 Determination of calcium from plaster of paris
- 14 Determination of total cation concentration in waste water sample by cation exchange resin
- 15 Determination of anion exchange capacity of anion exchange resin.
- 16 Analysis of water with respect to sulphate and chloride
- 17 To determine phosphoric acid in cold drink by molybdenum blue method
- 18 Estimation of Cu and Fe(III) by spectrophotometric titration
- 19 Limit test: i) Iron from CaCO_3 ii) sulphate and chloride from paracetamol, Dextrose or any pharmaceutical preparation
- 20 Determination of alcohol from given sample by spectrophotometry
- 21 Analysis of Zn-chrome pigment for zinc and chromium.
- 22 Volumetric determination carbonate and bicarbonate
- 23 Determination of Si, Ti, Ca and fluoride from toothpaste
- 24 Determination of Cu, Fe, Zn and Mn from soil sample by AAS method.
- 25 Synthesis of semiconducting nano oxides such as Fe_2O_3 .
- 26 Determination of phosphate in detergent by UV spectrophotometry
- 27 Determination of magnesium from milk of magnesia.
- 28 Magnetic susceptibility: MnSO_4

(Note: Minimum 16 experiments should be completed in this course.)

Report on industrial visit or study tour.

References:

1. Lab manual: selected experiments of Pharmaceutical analysis, Anees A Siddiqui.
2. Experiments in chemistry, D.V.Jahagirdar.
3. Pharmacopeia of India
4. Vogel's textbook of quantitative chemical analysis, sixth Ed.
5. Environmental chemistry by A.K.De.
6. Biochemical methods, Sadashivam and Manickem, Narosa publication
7. Quantitative inorganic analysis: Elementary Instrul. Analysis A. Vogel, 3rd ed. ELBS

Practical course II

CHA-5306: Instrumental Analysis (4 Credit)

- 1 To determine amount of each p-nitrophenol from the given sample by spectrophotometric titration.
- 2 Determination of strength of phosphoric acid by potentiometric titration using standard solution of sodium hydroxide.
- 3 Determination of commercial vinegar by potentiometric titration.
- 4 Analysis of riboflavin from vitamin supplementary capsules /syrup/tablet sample by photoflurometry.
- 5 Determination of Na and K from water sample by flame photometry binary method/ internal standard method.
- 6 Determination of boric acid by conductometry.
- 7 Determination of relative strength of acetic acid, chloroacetic acid and trichloroacetic acid through measuring their K_a value by conductivity measurement method.
- 8 Determination of amount each copper and bismuth or copper and iron(III) from given mixture by spectrophotometric titration using standard EDTA solution.
- 9 Determination of glucose saline sample by polarimetrically.
- 10 Determination of purity of sugar sample by optical rotation by polarimetry.
- 11 Estimation of heavy metals from waste water sample by AAS.
- 12 Analysis of paracetamol by HPLC.
- 13 Photometric determination of aluminium from given antacid tablet by working curve method
- 14 TGA for a mixture of CuSO_4 and NaCl . Find out the percentage of each constituent in the mixture.
- 15 P^{H} metric titration of anthranilic acid and glycine with NaOH .
- 16 Analysis of rock salt for chloride content by conductometric titration.
- 17 Analysis of alcohol from wine by GC.
- 18 Analysis of paracetamol/caffeine/metformin hydrochloride and nimucelide by HPLC
- 19 Determination of sulphate and chloride and turbidimetric method
- 20 Determination of copper and zinc in brass alloy by polarography.
- 21 Determination of Ranitidine content in tablet.

(Note: Minimum 16 experiments should be completed in this course)

References:

1. Lab manual: selected experiments of Pharmaceutical analysis, Anees A Siddiqui.
2. Experiments in chemistry, D.V.Jahagirdar.
3. Pharmacopeia of India
4. Vogel's textbook of quantitative chemical analysis, sixth Ed.
5. Environmental chemistry by A.K.De.
6. Biochemical methods, Sadashivam and Manickem, Narosa publication
7. Senior practical physical chemistry. B.D. Khosla and V.S. Garge (R.Chand and Co).
- 8.** Analytical chemistry by Gary Christian, 6th edition, 2008

M. Sc. - II Analytical Chemistry

Semester – IV

CHA-5401: Forensic science (48 L+ 12 T) (4 Credit)

1. Forensic analysis:

Overview, destructive and non-destructive techniques, data interpretation. (2 L)

2. Blood analysis:

Blood preservation and aging effects, analysis of blood components and exogenic substances, blood stain analysis. (3 L)

3. DNA Profiling:

DNA and its polymorphism, DNA typing procedures- RFLP, PCR, MVR-PCR, Dot –Plot, AMP-FLP, STR, other methods, paternity testing, applications, interpretation and practical use. (4 L)

4. Determination of alcohol in body fluids:

Legal background, sampling and sample preservation, analysis G-,IR, enzymatic and other methods. (4 L)

5. Fingerprint analysis:

Latent fingerprints, optical, physical, physico -chemical and chemical detection methods, fingerprints in blood ,fingerprint detection sequences. (4 L)

6. Hair analysis:

Structure and composition of hair, morphological examination, chemical analysis of hair components and components remaining on or in hair. (5 L)

7. Systematic Drug Identification:

Classification and categories of compounds involved, analytical-strategy-EMIT, FPIA, TLC, LC, GC-MS etc., requirements for identification, possibilities and limitations of selected techniques, isotope detection method with numerical, new drug groups. (6 L)

8. Materials of interest for forensic studies:

Explosives: Types, analytical methods for identification of low and high explosives in post- blast debris. **Fibers:** Fibers encountered at cyber scene, identification of type, dye extraction and analysis, color matching, analysis for metals, additives and contaminants- SEM-EDX, XRD and XRF.

Paints, Varnishes and Lacquers: Formulation of paints, types of samples, sample pre-treatment prior to analysis , colour measurements, Analysis by SEM, SEM-SPMA, TEM-TLC/HPTLC, PyrGC, IR, Raman , UV-VIS-Flu, XRF, AES, TG-DTA.

Glass: As forensic evidence, measurement of physical properties, elemental analysis- XRD, NAA, interpretation of results, casework examples.

Arson residue: Nature of evidence, chemical evidence, properties of liquid accelerants, sampling and sample pre-treatment, laboratory examination of suspect arson evidence, evidential value.

Gunshot residues: Composition of sources, detection on hands and its limitation, determination of muzzle-to-target distance, elemental and inorganic analysis, numerical on estimation of energy released by combustion reactions, (20 L)

References:

1. 'Forensic chemistry' by Suzanne Bell, Pearson Prentice Hall Publishers, 2006
2. Encyclopaedia of Analytical Chemistry, Volume 3, Academic press, 1995

CHA-5402 Advances in Analytical Techniques

(48 L+ 12 T) (4 Credit)

Section I- Analytical Extraction Techniques (24 L+ 06 T) (2 Credit)

1 Classical approach for aqueous extraction

Introduction, Liquid-Liquid extraction (LLE) (Theory of LLE, selection of solvents, solvent extraction, problems with LLE process), purge and trap for volatile organics in aqueous samples **(6 L, Ref 8)**

2 Solid Phase extraction (SPE)

Introduction, Types of SPE media, SPE formats and apparatus, method for SPE operation, solvent selection, factors affecting SPE, selected methods of analysis for SPE, Automation and On-Line SPE **(6 L, Ref 8)**

3 Solid phase micro-extraction

Introduction, theoretical considerations, experimental, Methods of analysis: SPMEGC, Methods of analysis: SPME-HPLC-MS, Automation of SPME, New development in micro extraction (liquid micro extraction, membrane micro extraction) **(6 L, Ref 8)**

4 Microwave assisted extraction

Introduction, instrumentation, Applications **(3 L, Ref 8)**

5 Supercritical fluid extraction

Introduction, instrumentation, Applications **(3 L, Ref 8)**

Section II- Atomic spectroscopic analysis (24 L+ 06 T) (2 Credit)

1. Atomic Spectroscopy:

Theory, sources, burners, atomic emission spectra, atomic absorption spectra, effect of temperature on emission, absorption and fluorescence, electro thermal atomizers, Instrumentation for FES, radiation sources atomic absorption methods, instrumentation for AAS, spectral interferences, standard addition and internal standard method of analysis, comparison of atomic absorption and emission methods, inductively coupled plasma and direct current plasma emission spectroscopy, Cold vapour technique, Applications of AAS, AES and ICPAES, analysis of micronutrients like Mo, B, Cu, Zn essential towards the healthy growth of crops, fruits, determination of these micronutrients from soils, plants and fruits **(12 L, Ref 1-5)**

2. Atomic Mass Spectroscopy:

Features of atomic mass spectroscopy, Atomic weight in mass spectroscopy, mass to charge ratio, Types of atomic mass spectroscopy, mass spectrometers, transducer for mass spectroscopy, quadruple mass analyser, time of flight mass analyzer, Inductively coupled mass spectroscopy (ICPMS), Instrumentation for ICPMS, Atomic mass spectra and interferences, Applications of ICPMS **(6 L, Ref 3, 4)**

3. Atomic Fluorescence, Resonant Ionization and laser based-Enhanced Ionization:

Atomic Fluorescence Spectroscopy (AFS): Atomic fluorescence, apparatus for AFS, EMR source for AFS, LASERS, Cells for AFS, Plasmas, Wavelength selection for AFS, Detectors for AFS, Theory of AFS, Analysis with AFS, Interference with AFS.

Resonant Ionization Spectroscopy, Laser-enhanced ionization spectroscopy. (6 L, Ref 1)

References:

- 1) Introduction to Instrumental Analysis, R. D. Broun, Mc. Graw Hill (1987)
- 2) Instrumental methods of chemical analysis, H. Willard, L.Merrit, J.A. Dean and F.A. Settle. Sixth edition CBS (1986)
- 3) Fundamentals of Analytical Chemistry, D.A. Skoog, D.M. West and F.J. Holler, Saunders college publishing, 6th edition
- 4) Principles of Instrumental Analysis, Skog, Holler, Nieman, (Sixth Ed.)
- 5) Vogel's Textbook of Quantitative analysis 6th Ed.
- 6) Modern analytical techniques in the pharmaceutical and bio analysis Dr. Istvan Bak (Book Available Online).
- 7) Preparative chromatography Chrome Ed. book series, Raymond P. W. Scott (free e-book available on internet)
- 8) Extraction technique in analytical science, John R. Dean, Wiley (2009)
- 9) Practical HPLC method Development, Snyder, Kirkiand, Glajch, Wiley India Pvt.Ltd.

**CHA-5403 Analytical methods for Analysis of fertilizers, detergents,
Water, and Polymer, Paint and pigment
(48 L+ 12 T) (4 Credit)**

**Section I- Analysis of fertilizers, soap and detergents, and waste water
(24 L+ 06 T) (2 Credit)**

1. Analysis of Fertilizers:

Sampling and sample preparation, water, total nitrogen: Kjeldahl's method, total nitrogen by reduced iron method, urea nitrogen, total Kjeldahl's nitrogen methods and spectrophotometric method, , Ammonia nitrogen. Phosphorus: total phosphorus, available and non-available, alkali metric ammonium molybdophosphate method, water soluble phosphorous, citrate insoluble phosphate, Potassium: potassium by sodium tetra phenyl borate method, flame photometric methods **(6 L, Ref 1)**

2. Analysis of soaps and detergents:

General scheme of analysis, sampling, alcohol soluble materials, moisture and volatile matter, active ingredient and equivalent combined SO_3^{3-}

Tests for soaps: total fatty acids, fatty anhydride combined alkali, and anhydrous soap, Unspunified and unsaponifiable matter, Free alkali or free acid, titer test, Iodine value, saponification value, free glycerol,

Tests for synthetic detergents: Unsulfonated or unsulfated matter, ester SO_3 , Combined alcohols, total combined SO_3 , Alkalinity, chlorides, silicate, phosphate, borates,

UV spectroscopic analysis of detergents: Biodegradability of detergents, Determination of sodium alkyl benzene sulfonate, determination of sodium toluene sulfonate, determination of sodium xylene sulfonate, determination of germicides in soaps and detergents. **(8 L, Ref 1)**

3. Water pollution and analysis of polluted water:

Water pollutants, waste water treatment: domestic waste water treatment, aerobic treatment process, anaerobic treatment process, industrial waste water treatment, The purpose of chemical analysis, sampling of water, pH of water, specific conductance, determination of acidity and alkalinity, chemical oxygen demand, biological oxygen demand, dissolved oxygen, turbidity, determination of aluminium, arsenic, boron, cadmium, calcium, carbon dioxide, chloride, residual chlorine, chlorine demand, chromium, cyanide, total hardness, iron, lead manganese, Zn, methane, nitrate, nitrite, ammonia, nitrogen, phenols, phosphates, silica, sulphate, sulfide, anionic detergents, tannin and lignin. **(10 L, Ref 1 and 3)**

Section II- Polymer, Paint and Pigment analysis (24 L+ 06 T) (2 Credit)

1. Introduction to polymers:

Brief history to polymers, how polymers are made? classification of polymers **(2 L)**

2. Analysis and testing of polymers:

a) Chemical analysis of polymers:

X-ray diffraction analysis, thermal analysis, TGA, DTA.

b) Physical testing of polymers:

Mechanical properties, Fatigue testing, impact testing, tear resistance, hardness, abrasion resistance.

c) Thermal properties: Softening temperature, flammability.

- d) Optical properties: transmittance, colour, gloss, haze and transparency.
- e) Electrical properties: dielectric constant and loss factor, resistivity, dielectric strength, electronic properties.
- f) Chemical properties: resistance to solvents, vapour permeability, weathering. **(10 L)**

3. Measurement of molecular weight and size:

End group analysis, Colligative properties measurements, solution viscosity and Molecular size. **(4 L)**

4. Analysis of Paints and Pigment:

Introduction, test on the total coating, water content, separation of pigment binder and thinner of solvent type coating, separation of pigment binder, and thinner of latex paints, Identification of the binder, Identification of polymer resins and oils, Identification of plasticizer, Analysis of the vehicle, Identification and Analysis of pigments, Identification of inorganic pigments, Analysis of white and tinted pigments, outline of general procedure, HCL insoluble, Titanium dioxide, total lead, acid soluble Al and Fe, acid soluble calcium, total zinc, antimony oxide, total sulfate, total carbonate) analysis of colored pigments, Black pigments, other pigments, identification and analysis of thinners. **(8 L, Ref 1)**

References

- 1) Standard methods of chemical analysis, F.J. Welcher volume 3, part-B,.
- 2) Standard methods of water and waste water analysis A. K. De.
- 3) Environmental Chemistry, A. K. De
- 4) Textbook of polymer science F.W.Billmeyer 3rd edition (1994).
- 5) Principles of polymer systems by F. Rodrigue, Tata Mc Graw Hill, New Delhi.
- 6) Principles of polymer systems by P.J.Flory, Cornell University press, New York.
- 7) Polymer chemistry-an introduction Seymour-Carraher-Marcel Dekker. Inc. New York.
- 8) Polymer Science by V.R. Gowarikar, N.B. Vishvanathane, New Age publisher (1998)
- 9) Polymer Science by VasantGowarikar, Wiley Eastern New York (1998).
- 10) Principle of polymer science, Bahadur and shastri, Narosa publishing

CHA-5404: Method of Analysis and Applications (48 L+ 12 T) (4 Credit)

Section I- Geochemical & alloy Analysis (24 L+ 06 T) (2 Credit)

Methods and Principle of analysis, reactions involved , flow sheet and procedures. Problems based on analysis of each material

1. Analysis of Geological materials:

Dolomite (For silicate, Mg and Ca content), Ilmenite (for silicate, Ti and Fe content), Monazite (for rare earth metals), Hematite and Magnetite (silicate and Fe content), Pyrolusite (for silicate and Mn content) and bauxite (for Al and Silicate content).

(8 L, Ref 1 and 2)

2 Analysis of Alloys:

Stainless Steel (for Fe, Cr, Ni, Co, Cu, Mn, W, Si, V, Mo, Ti, Pb and Zr)
Bronze and Gun metal (for Cu, Sn), , Brass (for Cu, Zn, Sn, Pb), Solder (for Pb and Sn), Nichrome (for Fe, Ni, Cr), Analysis of nickel Silver (Sn, pb, Cu, Fe, Ni and Zn) and Aluminium based alloys (Al, Mg,).

(8 L, Ref 1 and 2)

3 Analysis of Soil:

i) Sampling, ii) Carbonate, Organic carbon, and organic matter, iii) Total nitrogen, ammonia and nitrates, iv) Total determination of major soil constituents by fusion analysis, v) silica and total combined oxides of iron, aluminium, and titanium, vi) Determination Ca, Mg, Na, K, phosphate, boron, Co, Cu, Zn, vii) Exchangeable cations vii) Cation exchange capacity, viii) chemical analysis as a measure of soil fertility.

(8 L, Ref 1)

Section II- Analysis of body fluid (24 L+ 06 T) (2 Credit)

1. Collection of Specimens:

Blood: Collection of Blood specimens, storage and preservation, Urine: Collection of Urine, physical characteristics of urea, preservation and storage, Faeces: Collection and preservation.

(2 L , Ref 3)

2. Analysis of Blood and urine:

Determination of blood and plasma glucose by glucose oxidase method, Determination of urine for glucose, Determination of ketone bodies in blood, Oral Glucose tolerance test, Determination of serum creatinin, estimation of serum bilirubin, Estimation of serum cholesterol, determination of blood hemoglobin, Urate: determination of serum urate, Determination of urea in urine by urease method and by direct colorimetry, Estimation of Na, K, Ca by flame photometry, inorganic phosphate by colorimetry. **(6 L , Ref 3)**

3. Determination of vitamins in body fluid:

Classification of vitamins with example, Each vitamin must be explained with respect to functions, deficiency diseases, daily requirement and analytical method

i) Retinol (determination of retinol and serum carotene in serum using TFA), Vitamin D3 (cholecalciferol), Vitamin E (Tocopherols, Determination of serum Tocopherols by spectrophotometry by dipyrindyl method), Vitamin B1 (thiamine determination by flurometry), Vitamin B2 (riboflavin, Photofluorometric method), Vitamin B6 (Pyridoxine, Fluorometric determination of Xanthuric acid), Nicotinic acid and Niacin: determination by fluorometry, Ascorbic acid (vitamin C) Volumetric method using 2,6 dichlorophenol method, colorimetric determination of leucocyte ascorbate.

(8 L , Ref 3 and 4)

4. Immunoanalytical Techniques:

Radioimmunoassay, its principle and applications, instrumentation for radio bioassay, clinical application of the radioimmunoassay of insulin, Estrogen and progesterone, receptor techniques of breast cancer. Enzyme linked immunosorbent assay (ELISA), Types of ELISA, principles, practical aspects, applications. **(6 L , Ref 3 and 4)**

5. Organ function tests:

Liver function tests and kidney function tests **(2 L , Ref 3)**

References

- 1) Standard methods of chemical analysis, F .J. Welcher Sixth Edition,.
- 2) Quantitative Inorganic Analysis including Elementary Instrumental analysis, A. I. Vogel, 3rd, ELBS, 1964.
- 3) Practical Clinical Biochemistry, Gowenlock, CBS published, 6th Ed.
- 4) Biochemical methods of analysis, S. Sadasivam and A. Manickam, Narosa Publication

Practical course III

CHA- 5405: Analysis of pharmaceutical, food and Bio- analytical samples (4 Credit)

1. Determination of % purity of sodium benzoate.
2. Determination of % purity of lactic acid.
3. Determination of Dapson content in Dapson tablet
4. Determination of sulphamethoxazole content in co-trimoxazole tablet.
5. Identification of given unknown drug by using TLC- caffeine, Ibuprofen, quinine, salicylic acid and amino acid.
6. Chromatographic separation and identification of sugars/ amino acid
7. Estimation of total carbohydrates by anthrone method.
8. Determination of saponification value of oil.
9. Determination of iodine value of oil.
10. Assay of thiamine from given sample.
11. Analysis of quinine sulphate from given tablet by photoflurometry.
12. Estimation of tannin from given sample.
13. Analysis of caffeine from given tablet as per IP with respect to identification, assay.
14. Estimation of protein from food sample by Lowry method.
15. Analysis of paracetamol as per IP with respect to Identification, ash and assay.
16. Estimation of Fe from syrup sample by spectrophotometric method.
17. Estimation of HMF from honey.
18. Estimation of reducing sugar from food sample by spectrophotometry.
19. Determination of total acidity in juice.
20. Determination of glucose from glucon D by titration with Fehling method.
21. Moisture content in pharmaceutical/food sample by Karl Fischer titration method
22. Determination of adulterant in milk.

(Note: Minimum 18 experiments should be completed in this course)

References:

1. Lab manual: selected experiments of Pharmaceutical analysis, Anees A Siddiqui.
2. Experiments in chemistry, D.V.Jahagirdar.
3. Pharmacopeia of India
4. Vogel's textbook of quantitative chemical analysis, sixth Ed.
5. Environmental chemistry by A.K.De.
6. Biochemical methods, Sadashivam and Manickem, Narosa publication
7. Senior practical physical chemistry. B.D. Khosla and V.S. Garge (R. Chand and Co. Delhi)
8. Practical pharmaceutical chemistry 4th Ed .Part -2, Beckette, Stenlake.
9. Practical clinical biochemistry, Harold Varley (4th edition) , CBS publishers and distributors, New Delhi-110002
10. Analytical chemistry by Gary Christian, 6th edition, 2008

Practical course IV

CHA- 5406: Project work (4 Credit)

This is mandatory for every student to undertake the project work on selected area of study under the guidance of project coordinator. Student must carry out entire experimental work within the stipulated time and present it briefly in the form of the dissertation at the time evaluations.