ಮಂಗಳೂರು ವಿಶ್ವವಿದ್ಯಾನಿಲಯ

MANGALORE UNIVERSITY

(Accredited by NAAC with 'A' Grade)

ಕ್ರಮಾಂಕ/No. MU/ACC/CR6/CBCS-PG(SLB)/2017-18/A2

ಕುಲಸಚಿವರ ಕಚೇರಿ
ಮಂಗಳಗಂಗೋತ್ರಿ - 574 199
ಕರ್ನಾಟಕ, ಇಂಡಿಯ
Office of the Registrar
Mangalagangothri - 574 199
Karnataka, India

ದಿನಾಂಕ/Date : 8/5/2017

NOTIFICATION

Sub: III & IV semester Choice Based Credit System syllabus of M.Sc. in Analytical Chemistry degree programme.

Ref: 1) This office Notification No. MU/ACC/CR7/CBCS-PG(SLB)/ 2016-17/A2, dated: 17-8-2016.
2) Academic Council decision dated 3-2-2017 vide Agenda No. 3:11 (2016-17)

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In continuation to this office Notification cited under ref. (1) above, the syllabus of III & IV semester M.Sc. in Analytical Chemistry degree programme which approved by the Academic Council at meeting held on 3-2-2017 is hereby notified for implementation with effect from the academic year 2017-18 and onwards (for students of 2016-17 batch and onwards).

REGISTRAR

To:

1) The Chairman of the Department concerned/ The Coordinator of the degree programme concerned.
2) The Principal of the college concerned.
3) The Registrar [Evaluation], Mangalore University.
4) The Chairman of the Board of Studies concerned.
5) The Superintendent [ACC], Office of the Registrar, Mangalore University.
6) Guard file.

Mangalore University

Department of Studies in Chemistry

M. Sc. Degree Programme

(CHOICE BASED CREDIT SYSTEM - SEMESTER SCHEME)

Syllabi for M.Sc., Courses in

ANALYTICAL CHEMISTRY

(From the Academic Year 2016-17 onwards)

Programme : M.Sc., in Analytical Chemistry

3rd Semester		4th Semester	
CA H 501	Principles of Analytical Chemistry	CA H 551	Applied Analysis
CA H 502	Bioanalytical & Radiochemical Techniques	CA H 552	Optical Methods of Analysis
CA H 503	Chromatographic Separation Techniques	CA H 553	Process Analytical Chemistry
CA S 504 Or CA S 505	Thermoanalytical Techniques Or Analytical Chemistry of Polymers	CA H 554 Or CA S 555	Environmental Chemistry Or Pharmacokinetics & Nanimaterials
CA E 506	Analytical and Green Chemistry	CA S 555	Separation Techniques
CA P 507	Analytical Chemistry Practicals-III	CA P 557	Analytical Chemistry Practicals-VI
CA P 508	Analytical Chemistry Practicals -IV	AC P 558	Analytical Chemistry Practicals-VII
CA P 509	Analytical Chemistry Practicals -V	CA P 559	Project Work & Dissertation

3rd SEMESTER

CA H 501: PRINCIPLES OF ANALYTICAL CHEMISTRY

UNIT- I: Acid-base Titrations [15 Hours]

Titration curves for strong acid- strong base, weak acid- strong base and weak base- strong acid titrations. Poly protic acids, poly equivalent bases. Determining the equivalence points- theory of acid- base indicator, colour change range of indicator, selection of proper indicator, indicator errors. Feasibility of acid- base titrations- magnitude of the equilibrium constants, effect of concentration. Typical application of acid- base titrations. Determination of nitrogen, sulphur, ammonium salts, nitrates and nitrites, carbonates and bicarbonates and organic functional groups like carboxylic acid, sulphonic acid, amine, ester, hydroxyl, carboxyl groups , air pollutants.

Titrations in non- aqueous solvents- Solvents for non- aqueous titrations, characteristics of amphiprotic solvents- Autoprotolysis, Dielectric constant. Meaning of pH. Aprotic solvents, choosing a solvent. Some selected solvents, titrants and standards. Titration curves, effect of water. Determining the equivalence point. Typical applications- Determination of carboxylic acid, phenols, and amines.

UNIT- II [15 Hours]

Complexometric titrations: Complex formation reactions. Stability of complexes, stepwise formation constants, chelating agents. EDTA- acidic properties, complexes with metal ions, equilibrium calculations involving EDTA, conditional formation constants, derivation of EDTA titration curves, effect of other complexing agents, factors affecting the shape of titration curves. Completeness of reactions, indicators for EDTA titrations, theory of common indicators. Titration methods employing EDTA- direct, back and displacement titrations, indirect determinations. Titration of mixture, selectivity, masking and demasking agents, Typical applications of EDTA titrations- hardness of water, magnesium and aluminium in antacids, magnesium, manganese and zinc in a mixture. Titrations involving unidentate ligands- titration of chloride with Hg^{2+} and cyanide with Ag^{+}.

Precipitation titrations: Titration curves feasibility of precipitation titrations. Factors affecting shape- titrant and analyte concentration. completeness of the reaction. Titrants and standards. Indicators for precipitation titrations involving silver nitrate. The Volhard, the Mohr and the Fajan's methods. Typical applications.

UNIT -III: [15 Hours]

Redox titrations: Equilibrium constants for redox reactions-electrode potentials in equilibrium systems, calculation of equilibrium constants. Redox titration curves- formal potentials, derivation of titration curves. Factors affecting the shape of titration curves-concentration, completeness of reaction. Titration of mixtures-feasibility of redox titrations. Detections of end point-redox indicators, theory, specific and non specific indicators, choice of indicator, potentiometric end point detection. Sample preparation-prereduction and preoxidation. Karl Fischer reagent for water determination. Applications.

Gravimetric methods: Introduction to gravimetric analysis, precipitation methods, the colloidal state, Supersaturation and precipitate formation, purity of the precipitate: co-precipitation, post-precipitation, Conditions for precipitation, precipitation from homogeneous solution, washing the precipitate. Fractional precipitation, organic precipitants, volatilisation or evolution methods.

References:

1. Fundamentals of Analytical Chemistry-S.A.Skoog, West & Holler
2. Chemical Analysis- H.A Laitenin, Mc Graw Hill(1960)
3. Quantitative analysis-Kenner & Busch
4. Analytical Chemistry-Larry G. Hargis
5. Vogel's Text book of Quantitative Inorganic Analysis- Bessett, Denney, Jeffery & Mendham
6. Non-aqueous titrations-Waiter Huber, Academic Press(1987)
7. Quantitative analysis (5^{th} Edition)-R.A Day and A.L Underwood: P H I ,1988.

CA H 502: BIOANALYTICAL and RADIOCHEMICAL TECHNIQUES

UNIT- I: Bioanalytical Techniques -1 [15 Hours]

Membrane Electrodes: Classifications and properties, principle, Membrane potential, Sensors-types: crystalline, liquid membrane and enzyme electrodes, Gas sensors, voltammetric sensors, optical sensors, thermal sensors. **Biosensors:** Introduction bio-sensors- characteristics of an ideal biosensors. Basic electrochemical principles, measurement system. Enzyme based electrochemical bio sensors – theory and applications of glucose, urea and alcohol biosensors. Living biosensors (microbial, yeast based and botanical biosensor). Transduces technology, Enzyme based calorimetry, Enzyme reactors with HPLC. Fabrication and miniaturization: Glass micropipettes, Enzyme based micro electrodes and semiconductor needle.
Ion-Sensors: Ion selective electrode: Introduction, Types - Glass membrane electrodes, solid state ion exchange electrodes, solid state crystal electrodes, liquid membrane electrodes, and gas sensing electrodes. Analytical and biological applications of sensors.

UNIT- II: Radio Chemical methods of analysis [15 Hours]

Introduction, nature of radio activity, radiometric units, detection and measurements of radio activity. A brief introduction to radiolysis of gases , liquids and solids. Technique for study of transient species-Pulse radiolysis. Industrial applications of radiation chemistry (radiation synthesis, polymerization and food irradiation.
Health and safety aspects: Biological effects of radiation, Hazards in radiochemical work, Radiation protection, permissible exposure doses. Radioactive waste management.

UNIT-III : Radio Chemical methods of analysis (Radio analytical techniques) [15 Hours]

Disintegration theory, rate of disintegration, radioactive tracers, tracer techniques. Application in analytical chemistry, isotopic dilution analysis, activation analysis and prompt gamma neutron activation analysis (PGNAA). Radiometric analysis, radiometric titrations and applications.

Radio immuno assay, measuring radioisotope activity, antigens and antibodies, enzymatic immunoassay, its principle and applications, clinical application of the radioimmunoassay of insulin, Estrogen and progesterone, instrumentation for radiobioassay.

References:

1. Biosensor-Theory & Applications, Donald G. Burek,(Technomic Publication, Lancaster) 1993
2. Biosensors, ISHA Books, Delhi , Ed: Rajmohan Joshi, 2006
3. Bio Analytical Chemistry, Andreas Manz and Nicole Pamme, Imperial College Press, 2012
4. Bioanalytical Chemistry, Susan R. Mikkelson, Eduardo Colton, Wiley-Interscience, 2004.
5. Chemical Analysis, Francies Rouessac & Annick Roussac, 7th ed.,John Wiley, Chechester, 2007.
6. Separation Methods, Himalaya Publishing, Delhi, 2nd ed., M N Sastri 1996.
7. Principles of Instrumental Analysis, 5th ed., Skoog, Hollas and Nifman, Thomson Book, 2006.
8. Nuclear and Radio Chemistry- Friedlander, Kennedy and Miller.
9. Principles of radiochemistry- D.D Sood, N.Ramamoorthy and A.V.R Reddy
10. Instrumental methods of chemical analysis- B. K Sharma, 19th edition, Goel,2000

CA H 503: CHROMATOGRAPIC SEPERATION TECHNIQUES

UNIT-I: Basic concepts [15 Hours]

General description, definitions, terms and parameters used in chromatography. Classification of chromatographic methods. Criteria for selection of stationary and mobile phase-nature of adsorbents,factors influencing adsorbents, nature and types of mobile phases. Theoritical principles of chromatographic technique, development of chromatogram.

Elution Chromatography: theories-plate theory, rate theory. Band broadening-eddy diffusion, longitudinal, diffusion and resistance to mass transfer. Column efficiency-plate theory, rate theory approach. Van Deemters equation and its modern version. Inter relationships-capacity factor, selectivity factor, column resolution. Qualitative and Quantitative analysis by chromatography. Types of Chromatography.

UNIT-II: Planar chromatography and separation techniques [15 Hours]

Paper Chromatography (PC): Definitions , theory and principle. Migration parameter, Types of paper chromatography, Techniques; one, two-dimensional and circular PC. Mechanism of separation, structure of cellulose and types of paper. Methodology- preparation of sample, choice of solvents, location of spots and measurement of RF value. Factors affecting RF values. Experimental details for quantitative and qualitative analysis, Advantages and applications.

Thin layer Chromatography (TLC): definition, mechanism, efficiency of TL plates. Methodology – selection of stationary and mobile phases-preparation of plates, spotting, development, identification and detection. Reproductivity of RF values. Comparison of TLC with PC & Column chromatography. Qualitative and quantitative analysis. Applications and limitations of TLC.

UNIT-III: Column Chromatography [15Hours]

Conventional Column Chromatography - Principles & applications. High Pressure Liquid Chromatography (HPLC): Apparatus, pumps, column packing, characteristics of liquid chromatographic detectors-UV, IR, refractometer & fluorescence detectors. Advantages & applications.

Ion-exchange Chromatography (IEC): definitions , requirements for ion exchange resin. Synthesis and types of ion-exchange resins. Principle, basic features of ion-exchange reactions. Resin properties-ion-exchange capacity resin selectivity & factors affecting the selectivity. Applications of IEC in preparative, purification & recovery processes.

References:

1. Chromatography-E. Heftman(Ed), Part A and Part B, 5th ed.Elsevier, 1992.
2. Chromatography Today- D.F.Poole and S. K .Poole,.
3. Principles of Instrumental Analysis- Skoog, Holler and Nieman,5th ed. Saunders,1998
4. Quantitative Analysis – R.A.Day and A.L. Underwood, 5th ed.Prentice-Hall.1998
5. Instrumental Methods of Chemical Analysis- B.K.Sharma.19th.Goel, 2000

CA S 504: THERMO & SPECTRO ANALYTICAL TECHNIQUES

UNIT- I: Thermoanalytical Techniques [12 Hours]

Introduction, thermogravimetric analysis (TGA), types of thermogravimetric analysis, principle and method. Automatic thermogravimetric analysis, instrumentation, types of recording thermobalances, sample holders, factors affecting results and applications. Isothermal analysis. Application - purity and thermal stability, evaluation of correct drying temperature, analysis of complex mixture and determination and kinetic parameters of thermal degradation.

UNIT- II: [12 Hours]

Differential thermal analysis (DTA), principle of working, theory and instrumentation. Simultaneous DTA-TGA curves, factors affecting results, and applications. Analysis of physical mixtures and thermal behaviors study. Determination of melting point, boiling point and decomposition point. Differential scanning calorimetry (DSC) principles-Instrumentation and applications. Thermometric titration: introduction, apparatus and applications (Acid-Base, precipitation, Complexation, redox and non- aqueous titrations).

UNIT –III [12Hours]

Spectro analytical Methods

Colorimetric: Beer and Lambert's law – terminology – condition for a satisfactory colorimetric analysis – method of colour measurement or comparison – principles of colorimetric determinations of NH_3, Cr, Cu, Fe, Mn – simultaneous spectrophotometer determination of Cr and Mn.

^{1}H NMR and 13 C NMR elucidation of pharmaceutical drugs and its application with mass spectrophotometric studies. Use of electron spectroscopy, X-ray emission and Liminescence in analytical studies.

References

1. Fundamentals of Analytical Chemistry –S.A. Skoog, West & Holler
2. Instrumental Methods of Chemical Analysis; B.K. Sharma, 19th ed, Goel, 2000.
3. Principles of Electroanalytical methods, T.Riley and C.Tomlinson, Wiley, N.Y.,1987.
4. Principles of Instrumental Analysis-D.A. Skoog, 5th ed. Saunders College Pub. 1998
5. Quantitative Analysis (5th Edition) –R.A. Day and A. L. Underwood: P H I, 1988.
6. Quantitative analysis-C. T. Kenner and Kenner W. Busch
7. Vogel's Text book of Quantitative Inorganic Analysis-Bessett,Denney, Jeffery & Mendham
8. Analytical Chemistry- Larry G. Hargis .
9. Chemical Analysis - H.A. Laitenin, Mc Graw Hill (1960). D.A.Skoog, D.M.West and F.J.Hollar.
10. Fundamentals of Analytical Chemistry, 7th Edition, Harcourt College Publishers, 1996.
11. H.H.Williard. L.L.Merritt and J.A.Dean, Instrumental Methods of Analysis, East-West press, New Delhi, 1988.
12. J.G.Dick, Analytical Chemistry, Tata – Mc-Graw Hill, l973.

CA S 505: ANALYTICAL CHEMISTRY OF POLYMERS

UNIT- I: **[12 Hours]**

Basic concepts-Monomers, repeat units, degree of polymerization. Polydispersion, average molecular weight and size concept-number average, weight average, viscocity average and Z-average molecular weights. Average end to end distance and radius of gyration, Practical significance of molecular weight, Geometrical structure. Glass transition and melting transitions, Effect of polymer structure on Tg and Tm temperature,Tg -transitions and associated properties, importance of Tg, Effect of crystallinity on the properties of polymers and Tg.

UNIT -II: **[12 Hours]**

Advanced polymeric materials: Polymer blends interpenetrating Networks & composites – Types, preparation techniques, properties & application.

Polymer Processing – Processing of plastics, elastomers & fibres. Compounding & processing techniques-calendering, casting moulding foaming, fibre spinning & reinforcing techniques.

Identification of commonly used thermoplastics (ABS, Teflon, Acrylics, PE, PP, PVC,) an thermosets (expoxy and formaldehyde based polymers).

Environmental stability and testing-Photo, oxidative and biodegradation. Solvent resistance testing. Polymer additives & roles-fillers, plasticizers, antioxidants, coloring agents, UV stabilizers, heat stabilizers and flame retardants.

UNIT III: **[12 Hours]**

Determination of molecular weight– End group analysis, viscosity, light scattering, osmometry, cryoscopy, ebulliometry and ultra centrifugation method. **Thermal Characterization:** Thermal properties: Techniques of measurement of transition temperatures and stability, Dilatometry, DSC, DTA, DTG, TGA. Measurement of thermal conductivity, thermal expansion and heat deflection temperature.

Mechanical Properties – Tensile impact and flexural strengths. Deflection temperature. Dynamic mechanical thermal analysis and Flammability test-limiting oxygen index. Electrical properties: Measurement of dielectric strength, dielectric constant and arc resistance. Characterization and structural property relationship of polymers(phthalocyanine polymers, conducting polymers, flame retardant polymers, bio-medical polymers) with special interest. Special techniques-UV, IR, NMR, ESR, X-Ray Diffraction and Scanning Electron Microscopy methods of analysis.

References:

1.Polymer characterization, D. Campbell and J.R . White, Chapman and Hall, New York.
2.Textbook of Polymer Science, F.W . Billmeyer, Wiley, Newyork;
3.Polymer Science and Technology, J,R Fried, Prentiecer Hall, New York.
4.Fundamental Principles of Polymer materials, S.L Rosen, Wiley, New York.
5.A text book of Polymer science, P.L Nayak and S. Lenka, Kalyani, Ludhiana.
6.Functional Monomers and polymers, K. Takemoto, Y. Lanki and R.M Oettenbrite
7.Plastics- Chemistry and technology, W.E. Driver
8.Introductory Polymer chemistry. –G.S Misra, Willey Eastern, Newdelhi.
9.Polymer Composite Materials: Engineering & science Mathews & Rawlings (Chapman & Hall).
10.Polymer Science – V.R Gowrikar, N.V, Viswanathan and J. Sridhar, Wiley Eastern

CH E 506 : ANALYTICAL AND GREEN CHEMISTYRY

UNIT- I: **[12 Hours]**

UV/Electronic Spectroscopy: Basic principles, Beer-Lambert law, types of absorption bands, Factors affecting the positions of UV bands. Theoretical prediction of λmax for polyenes, α,β-unsaturated aldehydes, ketones (Woodward-Fieser rules) and substituted benzenes.

IR Spectroscopy: Basic principles, Application of infrared spectroscopy in the structural study-identity by finger printing and identification of functional groups. Characteristic vibrational frequencies of alkanes, alkenes, alkynes, aromatic compounds, alcohols, ethers, phenols and amines). Study of vibrational frequencies of carbonyl compounds (ketones, aldehydes, esters, amides and acids). Factors affecting band positions and intensities

Nuclear Magnetic Resonance Spectroscopy: Basic principles, Solvents used, chemical shift and its measurements, factors affecting chemical shift. Integration of NMR signals, spin-spin coupling, coupling constant. Shielding and deshielding. High resolution ^{1}H NMR. Applications of NMR spectroscopy in structure elucidation of simple organic molecules.

Mass Spectrometry: Basic principles, molecular ions, meta-stable ions and isotope ions. Fragmentation processes, McLafferty rearrangement. retro Diels-Alder fragmentations. Nitrogen rule.

UNIT- II: **[12 Hours]**

Hydrologic cycle, sources, chemistry of sea water, criteria and standards of water quality- safe drinking water, maximum contamination levels of inorganic and organic chemicals, radiological contaminants, turbidity, microbial contaminants. Public health significance and measurement of colour, turbidity, total solids, acidity, alkalinity, hardness, chloride, residual chlorine, sulphate, fluoride, phosphate and different forms of nitrogen in natural and polluted water. Chemical sources of taste and odour, treatment for their removal, sampling and monitoring techniques. Determination and significance of DO, BOD ,COD and TOC. Water purification for drinking and industrial purposes, disinfection techniques, demineralization, desalination processes and reverse osmosis . Treatment of liquid radioactive wastes

UNIT- III: **[12 Hours]**

Green Chemistry: Definition and principles, planning a green synthesis in a chemical laboratory, Green preparation-Aqueous phase reactions, solid state (solventless) reactions, photochemical reactions, Phase transfer catalyst catalysed reactions (Quaternary ammonium salts & Crown ethers), enzymatic transformations & reactions in ionic liquids.

Sonochemistry: Introduction, instrumentation, the phenomenon of cavitation, Sonochemical esterification, substitution, addition, oxidation, reduction and coupling reactions.

Microwave induced organic synthesis: Introduction, reaction vessel and reaction medium, concept, specific effect, atom efficiency, % atom utilisation, advantages and limitations, alkylation of active methylene compounds, N-alkylation, condensation of active methylene compounds with aldehydes, Diels-Alder reaction, Leuckardt reductive amination of ketones, ortho ester Claisen rearrangement.

References:

1. Organic Spectroscopy-3rd Ed.-W.Kemp (Pagrave Publishers, New York), 1991.
2. Spectrometric Identification of Organic Compounds - Silverstein, Bassler & Monnill (Wiley)1981.
3. Applications of Absorption Spectroscopy of Organic Compounds-Dyer(Prentice Hall,NY) 1965.
4. Spectroscopy of Organic Compounds-3rd Ed.-P.S.Kalsi (New Age, New Delhi) 2000.
5. Spectroscopic Methods in Organic Chemistry - Williams and Fleming, TMH.
6. A.K. De : Environmental Chemistry, (Wiley Eastern).
7. S.K.Banerji : Environmental Chemistry, (Prentice Hall India), 1993.
8 S.D. Faust and O.M. Aly : Chemistry of Water Treatment, (Butterworths),1983.
9. Sawyer and McCarty, Chemistry for Environmental Engineering(McGraw Hill) 1978
10. I.Williams, Environmental Chemistry, John Wiley, 2001.
11. S.M.Khopkar, Environmental Pollution Analysis, (Wiley Eastern).

12. Organic Synthesis-Special Techniques, V.K.Ahluwalia & R. Aggarwal, Narosa, 2001.
13. Green Chemistry-Environment friendly alternatives- R.Sanghi & M.M.Srivatsava, Narosa, 2003.
14. Green Chemistry-Environment benign reactions- V.K.Ahluwalia, Ane Books India, 2006.

CA P 507: ANALYTICAL CHEMISTRY PRACTICAL – III

1. Analysis of Na_2CO_3 and $NaHCO_3$ in baking soda by acid base titration
2. Determination of Calcium in limestone by redox titration
3. Determination of total hardness of water by EDTA titration
4. Analysis of copper/Calcium by PFHS method
5. Analysis of chloride in natural and polluted water samples by argentimetric titration
6. Assay of calcium in milk powder by EDTA titration
7. Assay of Vitamin – C by iodine titration and by bromination using $KBrO_3$
8. Determination of iron and sodium in water by spectrophotometry.
9. Assay of iron in pharmaceutical preparation by visual & potentiometric titration by $Ce(SO_4)_2$
10. Iodometric determination of antimony.
11. Quantitative analysis of mixtures: a) Chloride and iodide; b) iodide volumetrically using KIO_3 & ii) total halide gravimetrically
12. Analysis of Calcium and lead-using EDTA

CA P 508: ANALYTICAL CHEMISTRY PRACTICALS – IV

1. Estimation of iron in razor-blade by potentiometric & visual titration using sodium vanadate.
2. Determination of water in hydrated calcium sulphate by Karl-Fischer titration
3. Determination of chloride content in tap water by Spectrophotometry.
4. Estimation of sulphate in urine by precipitation titration following ion-exchange separation.
5. Determination of 1, 2-glycols or glycerol by periodates oxidation.
6. Determination of aluminium and magnesium in antacids by EDTA Titration
7. Determination of mercury in algaecide by EDTA titration
8. Determination of Iron in mustard seed by spectrophotometry
9. Extractive Spectrophotometric determination of lead in leaf.
10. Determination of copper by potentiometric titration using EDTA.
11. Conductometric determination of total acidity of waste water.
12. Conductometric determination of chloride content of a sample of industrial effluent
13. Conductometric determination of total alkalinity of waste water.
14. Determination of pH of hair shampoos by potentiometric titration.

CA P 509: ANALYTICAL CHEMISTRY PRACTICALS –V

1. Analysis of alloys: Solder – lead and tin using EDTA.
2. Analysis of Copper-Nickel alloy i) Copper volumetrically using KIO_3 & ii) Nickel gravimetrically using DMG
3. Analysis of Brass - Cu gravimetrically using α- benzoinoxime and Zn complexometrically
4. Analysis of Stainless steel-Ni gravimetrically using DMG, Fe volumetrically using Ce(IV), Cr volumetrically by persulphate oxidation,
5. Paper chromatographic separation of i) iron and nickel; ii) copper and nickel
6. Separation and determination of chloride and bromide on an ion-exchanger.
7. Estimation of total cation concentration in water by ion-exchange method.
8. Determination of nickel in Cu-Ni alloy by electrogravimetry.
9. Analysis of copper in ore/alloy by iodometric titration
10. Analysis of ores – chalcopyright, ilmenite.
11. Analysis of Glass and ceramics.
12. Computational Chemistry Laboratory: Windows and Linux; MSOFFICE; Statistical Data Processing and Curve Fitting by EXCEL, GRAPHER, SURFER and MATHEMATICA; Chemical Structure Drawing by ISIS Draw, CHEMWIND, ACD Labs and CHEMDRAW; Molecular Modeling by ACD Labs, PCWIN and CHEM 3D; Chemical Databases; Animations and Virtual Chemical Experiments.

References:

1.A text Book of Quantitative Inorganic Analysis – A.I Vogel
2.Vogel's Text Book of Quantitative inorganic Analysis, Basset, Denney, Jeffery & Mendham
3.Colorimetric Determination of Traces of Metals – E.B Sandell.

4th SEMESTER

CA H 551: APPLIED ANALYSIS

UNIT- I: Drugs and Pharmaceutical Analysis [15 Hours]

Importance of quality control; drugs and pharmaceuticals. Sources of impurities in pharmaceutical chemicals. Analytical quality control in finished/final products. Common methods of assay.

Analysis of common drugs: Analgesics- aspirin, paracetamol, Antihelmentics-mebendazole; Antiallergies–chlorpheneramine malleate; antibiotics- pencilin, chloromecytin; Anti-inflammatory agents-Oxyphenbutazone; Antimalarials-primaquine phosphate; Antituberculosists-1 NH; Narcotics-nicotine, morphine; Expectrorants-benadryl; sedatives-diazepam; vitamins-A,B1, B2, B6, C, niacin & folic acid.

UNIT- II: Biomedical and forensic analysis [15 Hours]

Composition of body fluids and detection of abnormal levels of certain constituents leading to diagnosis of disease. Sample collection and preservation of physiological fluids, Analytical methods for the constituents of physiological fluids (blood, serum urine). Blood-Estimation of cholesterol, haemoglobin and bilirubin. Urine –Urea, Uric acid, creatinine calcium phosphate, sodium, potassium and chloride. Biological significance, analysis and assay of enzymes (pepsin, monoaminoxide); Vitamins (thiamine, vitamin A).

Forensic analysis: General discussion of poisons with special reference to mode of action of cyanide, organophosphates and snake venom. Estimation of poisonous (materials such as lead, mercury and arsenic in biological materials). Forensic Toxicology :Analysis of various types of poisons (corrosive, analgesic, hypnotic, narcotic, stimulants, paralytic, antihistamine).

UNIT III: Food Analysis [15 Hours]

General methods for the determination of moisture, ash, crude protein, fat, crude fibre carbohydrates, calcium, potassium, sodium and phosphates. Dairy products – composition of milk and milk products. Alcohol test, fermentation, dye reduction, methylene blue and resazurin tests. Analysis of fat content, minerals in milk and butter. Estimation of added water in milk. **Oils and fats-** General composition of edible oils, detection of purity, rancidity of fats and oil, estimation of rancidity, tests for common edible oils like groundnut oil, castor oil, cottonseed oil

and mustard oil. Tests for adulterants like aregemone oil and mineral oils. Significance of value of iodine value and saponification value of oils and fats.

Beverages – soft drinks, alcoholic drinks, tea, coffee and fruit juices. **Food additives, adulterants and contaminants** – food preservatives like benzoates, propionates, sorbates, bisulphites. **Artificial sweeteners** like saccharin, dulcin and sodium cyclamate. Flavours- Vanillin, esters (fruit flavours) and monosodium glutamate. Artificial food colourants- coal tar dyes and non-permitted colours and metallic salts. Pesticide residues in food.

References:

1.Chemical Analysis – H.A. Laitinin, Mc Graw Hill., (1960).

2.P.L. Kirk, Quantitative Ultramicroanalysis, John Wiley.

3 .G.D. Christian, Analytical Chemistry, John Wiley & Sons, New York (2001).

4. S.M. Khopkar, Analytical Chemistry of Macrocyclic and Supramolecular Compounds, Narosa Publishing House, New Delhi (2002).

5. Jag Mohan, Organic Analytical Chemistry - Theory and Practice, Narosa Publishing House, New Delhi (2003).

6.Pharmaceutical analysis – Ed. By T. Higuchi and E.B Hanssen, Wiley New York.

7.The Quantitative Analysis of Drugs – D.C Garratt, Chappman and Hall, New York

8.Pharmaceutical Analysis Modern Methods- Parts A and B, Ed. By James W. Munson.

9.Drugs and Pharmaceutical Sciences Series, Marcerl Dekkar, Vol. II INC, New York.

10.Analysis of Foods – H.E. Cox;

11.Chemical analysis of Foods – H.E Cox

12.Foods: Facts and Principles – N Shakuntala Many & S. Swamy, 4th ed. New Age International (1998).

13.The Essential of Forensic Medicine and Toxicology – K.S Narayana Reddy.

14.W. Horwitz, Official Methods of Analysis, 11th Edition (1970), Association of Official Analytical Chemists, Washington DC.

15. K. Simpson and B. Knight, Forensic Medicine, 9th Edition (1985), Edward Arnold Publishers Ltd., London.

CA H 552: OPTICAL METHODS OF ANALYSIS

UNIT –I: Atomic Spectroscopy [15 Hours]

Flame photometry-Emission and atomic absorption spectroscopy – Introduction, principle, flames and flame spectra, variation of emission intensity with the flame, flame temperature, chemical reactions in flame, metallic spectra in flame, flame background. Total consumption and premix burners, role of temperature on absorption, emission and fluorescence. Effect of organic solvents. Comparative study of the basic components and difference in the instrumental design for atomic absorption and flame photometry. Precision and accuracy of AAS and FES. Relationship between AAS and FES, advantages over FES, devices used for the formation of an atomic vapour, applications, determination of sodium in different samples by flame photometry.

UNIT -II: Molecular Luminescence [15 Hours]

Fluorometry and phosphorimetry **:**Introduction, fluorescence and phosphorescence, factors affecting fluorescence and phosphorescence, internal conversion, intersystem crossing (radiationless processes) quenching. theory, relationship between intensity of fluorescence and concentration, instrumentation– basic differences in the measurement of fluorescence and phosphorescence, filter fluorometers, spectrofluorometers, advantages, limitations and precautions. Selection of excitation wavelength for analysis, reporting fluorescence spectra. Application of fluorometric analysis of inorganic, organic, Special fluorometric and phosphorimetric applications. chemiluminescence.

UNIT-III: [15 Hours]

Nephelometry and Turbidimetry: Light scattering, nephelometry and turbidimetry, choice between nephelometry and turbidimetry, tubidimetry and colorimetry, nephelometry and fluoromtry. Theory effects of concentration, particle size and wavelength on scattering instruments and application of nephelometry and turbidimetry. Turbidimetric titrations.

Photoelectron spectroscopy: Introduction, instrument, source, target, electron energy analyzer, detector, technique and application of photoelectron spectroscopy in elemental, quantitative, gas and structural analysis. Study of core binding energy, charge distribution in bonds and molecules, surface chemistry and study of metal complexes.

References:

1. Principles of Instrumental analysis – Skoog, Holler and Nieman, 5th ed. Sauders, 1988
2. Analytical Chemistry: Principles – J.H Kennedy, 2nd ed. Saunder College Pub. 1990/
3. Quantitative analysis – R.A. Day and A.L Underwood, 5th ed. Prentice – Hall, 1998
4. Analytical Chemistry – G.D Christian, 4th ed, Wiley, 1986.
5. Chemical Analysis – An Instrumental Approach- Srivastava & Jain, S. Chand 1997
6. Modern Optical methods o Analysis – E.D Olsen, Mc Grow- Hill 1975.
7. Nanochemistry: A Chemical Approach to Nanomaterials -G.A. Ozin & A.C Arsenault, RSC publishing 2005.
8. Nanoscale materials-Ed. L.M. Liz-Marzan & P.V. Kumar (Kulwar) 2003.
9. Introduction to nanotechnology-C. P. Poole & J. F. Owenns (Wiley Interscience) 2006.

CA H 553: PROCESS ANALYTICAL CHEMISTRY

UNIT -I: Automation of Analytical Methods [15 Hours]

An overview of automated systems-Definition-Distinction between automatic and automated systems-Advantages and disadvantages by automation-Types of automated techniques. NM-discrete techniques, Segmented flow methods and basic equipment, special techniques and devices, theoretical considerations and problems. Applications, Single/channel and multi channel auto analysers, BUN analysers, automatic glucose analyzer, ammonia in water analyzer. COD analyzer, CFA in iodometry.

UNIT II: Non-segmented flow methods [15 Hours]

Flow injection analysis, principles, types of dispersion, factors affecting dispersion, application of low, medium and large dispersion. Stopped flow methods. Flow injection titrations. Discrete methods. Centrifugal fast scan analyzer, automatic multipurpose analyzer, automatic elemental analyzer. Automated analysis based on multilayer films – principles, film structure instrumentation, applications. Comparison of discrete and non-discrete methods, advantages of flow injection measurements over continuous flow measurements.

UNIT III: Good Laboratory Practice Terms [15 hours]

Safe working procedure and protective environment, protective apparel, emergency procedure and first aid, laboratory ventilation, safe storage and use of hazardous chemicals, procedure for

working with substance that pose hazards, flammable or explosive hazards – safe storage and disposal of waste chemicals, recovery, recycling and reuse of laboratory chemicals.

An introduction to quality control and quality assurance-Basic concepts, quality assurance, aspects of specification and tolerance, quality acceptance, sampling. reliability, cost aspects of quality decisions. Quality control in raw materials, production (in process), finished product. Current trends in quality control, ISO 9000 and ISO 14000 series. Laws related to quality control.

References:

1. Environmental Chemistry- Manahan, S.E, Lewis Pub., 4th ed., Columbia (1990)
2. Chemistry for Environmental Engineering – Sawyer and McCarty, 3rd Mc Graw Hill 1978.
3. Environmental Chemistry – A.K De;
4. Environmental Pollution Control and Engineering – C.S Rao, Wiley-Eastern, New Delhi, 1991;
5. Technical Methods of Analysis – R.C. Griggin
6 Principles of Instrumental Analysis –Skoog, Holler & nieman, 5th Ed., Saunders, 1998.
7 Analytical Chemistry: Principles – J.H Kennedy, 2nd ed., Saunders, 1990
8 Quantitative Analysis – Day and Underwood, Prentice- Hall, 1998;
9 Statistical Quality Control- M. Mahajan, Dhampath Rai & Sons, NewDelhi 1995;
10 Stoichiometry for Chemical Engineers – William and Jhonson.
11 Transport processes and Unit Operations C.J Geankopils, Prentice Hall (1977);
12 Manning : A Text Book of chemical Industry;

CA S 554: ENVIRONMENTAL AND PROCESS ANALYTICAL CHEMISTRY

UNIT- I: Soil and Fuel analysis [12 Hours]

Soil Analysis: . Preparation of laboratory sample. Measurement of pH and conductivity, acidic and alkaline soil. Analysis of major constituents-organic matter, nitrogen, sulphur, potassium and calcium. Analysis of trace elements –copper, molybdenum, zinc, boron. Analysis of major constituents-organic matter, nitrogen, sulphur, potassium and calcium.

Fuel Analysis: Defnition and classification of fuels, Characteristics of fuels.Sampling, Proximate and Ultimate analysis. Combustion calculations. Calorific value determination by Junker's gas calorimeter. Liqiud fuels- Determination of flash point, fire point, aniline point. Knocking of fuels Octane and Cetane numbers, Carbon residues.Gaseous fuels- Analysis of Coal, water gas, producers gas, gober gas and blast furnace gas. Calorific value determination by Junker's gas calorimetry

UNIT II : Air Pollution and Analysis and control [12 Hours].

Classification and properties of air pollutants. Emission sources, major emissions from global sources. Behaviour and fate of air pollutants –wet precipitation, dry deposition, interaction at the earth's surface, chemical reactions in the atmosphere, photochemical smog. Effects of air pollution on human health, vegetation and materials.

Analysis of air pollutants. SO_2 –ambient air measurements, stack gas measurement NOx – Griess-Saltzmann and Jacob-Hocheiser methods. CO-NDIR, amperometric, FID & catalytic oxidation methods. Oxidants & ozone-colorimetric, and chemiluminescent methods. Hydrocarbons-total and individual hydrocarbons by chromatographic methods. Particulates optical & mass measurement methods.

UNIT-III: Air Pollution control Methods [12 Hours].

Source Correction methods-raw materials changes, process changes, equipment modification or replacement. Particle emission control-gravitational setting chambers, cyclone collector, electrostatic precipitator, fabric filters and wet scrubbers.**Control of gaseous emissions. SO_2** – desulpharization of fuels, sulphur reduction during combustion and desulpharization of flue gases. **NOx** –modification of operating conditions, effluent gas treatment methods. **CO-** industrial sources and transportation sources. Hydrocarbons-incineration, absorption, adsorption and condensation methods, two-stage catalytic converters for mobile sources.

References:

1. Environmental Chemistry- Manahan, S.E, Lewis Pub., 4th ed., Columbia (1990)
2. Soils in our Environment: Raymond W. Miller, Duane T. Gardiner, Prentice Hall, 8th Ed.
3. Chemistry for Envi. Engineering – Sawyer and McCarty, 3rd Mc Graw Hill 1978.
4. Environmental Pollution Control & Engineering – C.S Rao, Wiley-Eastern, Delhi,1991.
5. Technical Methods of Analysis – R.C. Griggin
6. Principles of Instrumental Analysis –Skoog, Holler & nieman, 5th Ed., Saunders, 1998.
7. Analytical Chemistry: Principles – J.H Kennedy, 2nd ed., Saunders, 1990
8. Quantitative Analysis – Day and Underwood, Prentice- Hall, 1998;
9. Statistical Quality Control- M. Mahajan, Dhampath Rai & Sons, NewDelhi 1995;
10. Stoichiometry for Chemical Engineers – William and Jhonson.
11. Transport processes and Unit Operations C.J Geankopils, Prentice Hall (1977);
12. Manning : A Text Book of chemical Industry;

CA S 555: PHARMACO & NANOMATERIALS

Unit-I: Pharmacokinetics [I2 Hours]

Pharmacokinetics : Introduction – Compartment models –study of the methods of estimation, significance of the following parameters, biological half- life, apparent volume of distribution, renal clearance, total body Clearance, absorption rate, AUC - Mathematical expressions describing the variation in blood concentrations following I.V. and oral routes . Introduction to dosage regimen. **Non-linear Pharmacokinetics** : Non-linear Pharmacokinetics with special reference to one compartment model after IV drug administration, Michaeles-Menten equation.

Unit-II :Nano Materials-I [I2 Hours]

Introduction, Properties (size dependent) Synthesis- bottom up approach (sol- gel, precipitation, gas condensation, Chemical vapour condensation, hydrothermal & thermolysis process). Nano scale materials – nano crystals & clusters, nano crystalline fullerenes, carbon nanotubes, nano wires, nano rods, dendrimers & nano composites. Chemical synthesis of nanoparticles(chemical reduction, sol-gel technique and microwave synthesis), Functionalized nanoparticles in different medium. Size control, self assembly. Nanoparticles arrays. Analysis of nanostructure by AFM, STM, TEM and related techniques.

Unit-II: Nano Materials-II [I2 Hours]

Solution growth techniques of 1D-2D nano structures:- Synthesis of metallic, semiconducting and oxide nanoparticles – homo- and hetero-nucleation growth methods – template-based synthesis (electrochemical, electrophoretic, Melt and solution, CVD, ALD) – Gas Phase Synthesis of Nanopowders: – Vapor (or solution) – liquid – solid (VLS or SLS) growth – the Need for Gas/vapor State Processing – Main Stages of Gas Phase Synthesis – Applicability of the methods. Homogeneous Nucleation – Spinodal decomposition – Other undesirable Post-Condensation Effects – Nanoparticles' morphology

References:

1. Nanochemistry: A Chemical Approach to Nanomaterials -G.A. Ozin & A.C Arsenault, RSC publishing 2005.
2. Nanoscale materials-Ed. L.M. Liz-Marzan & P.V. Kumar (Kulwar) 2003.
3. Introduction to nanotechnology-C. P. Poole & J. F. Owenns (Wiley Interscience) 2006.
4. Nano-Forensic analysis to identify, individualize and evaluate evidence using nanophase materials
5. Scratch resistance enhancement study of polymer containing nanoparticles
7. Pharmacokinetics by Gibaldi
8. Biopharmaceuticals and Pharmacokinetics by R.E.Notari.
9. Pharmacokinetics by Ritschal
10. Modern Pharmaceutics by G.S.Banker
11. Applied Biopharmaceutics and Pharmacokinetics, Leon Shargel
12. Clinical Pharmacokinetics; Concepts and applications by T.Rowland and Tozer
13. Magnetic separation/identification studies of thermally-blocked nanoparticles

CA S 556: Separation Techniques

UNIT-I: **[12Hours]**

Solvent Extraction: Definition, types, principle and efficiency of extraction. Sequence of extraction process. Factors effecting extraction-pH, oxidation state, modifiers, synergistic, masking and salting out agents. Techniques-batch and continuous extraction. Application.

Affinity Chromatography: Definitions. separation mechanism- matrices, matrix activation, role of spacer arms and applications.

Electrophoresis: Theory and classification. Factors influencing the mobility-macromolecular size and charge, interaction with supporting electrolyte, pH and concentration discontinuities. Factors affecting electrophoretic phenomena-electrolysis, Electro osmosis, temperature and supporting media. Instrumentation. Methodology-preparation of gels-staining and destaining.

UNIT-II: **[12Hours]**

Exclusion Chromatography: Theory & principle of size exclusion chromatography . Experimental techniques for gelfiltration chromatography (GFC) & gel-permeation Chromatography (GPC).Materials for packing-factors governing column efficiency. Methodology & applications. **Gas Chromatography (GC):** Principle, comparison of GSC & GLC. Instrumentation Columns-packed and tubular. Study of detectors- thermal conductivity, flame ionisation, electron capture & mass spectrometry. Factors affecting separation. Applications of GC/MS technique.

UNIT-III: **[12Hours]**

Ultracentrifugation: Principle sedimentation constant, sedimentation equilibrium, sedimentation velocity, methodology and applications.

Capillary electrophoresis: Overview, types, the basis for electrphoretic separations, migration rates and plate heights, electro-osmotic flow, instrumentation, capillary zone electrophoresis, capillary gel electrophoresis, capillary isoelectrophoresis, capillary isoelectric focusing. **Capillary electrochromatography**: Packed column electrochromatography, micellar electrokinetic capillary chromatography and applications.

References:

1. Chromatography-E. Heftman(Ed), Part A and Part B, 5th ed.Elsevier, 1992.
2. Chromatography Today- D.F.Poole and S. K .Poole,.

3. Principles of Instrumental Analysis- Skoog, Holler and Nieman,5th ed. Saunders,1998
4. Quantitative Analysis – R.A.Day and A.L. Underwood, 5th ed.Prentice-Hall.1998.

5. Analytical Chemistry, G.D. Christian, 5th ed., 2001 John Wiley & Sons, Inc, India

CA P 557: ANALYTICAL CHEMISTRY PRACTICALS – IV

(A minimum of twelve experiments are to be carried out)

1. Potentiometric determination of equivalent weight and Ka of a pure unknown acid.
2. Nephelometric determination of sulphate in ground water samples.
3. Nephelometric determination of chloride in brackish waters.
4. Determination of Phosphate in domestic waste water by spectrophotometry.
5. Determination of Vitamin A in Vanaspathi by UV spectroscopy.
6. pH-metric determination of soda ash in washing soda..
7. Determination of sodium and potassium in soil by flame photometry.
8. Estimation of potassium in agricultural water supplies by flame photometry.
9. Determination of NH_3-N/NO_2-N/NO_3-N in soil & waste waters by spectrophotometry.
10. Fluorimetric determination of Quinines, cadmium aluminium and zinc.
11. Determination of sulpha-drugs by potentiometry
12. Evaluation of the composition of Fe (II)-phen complex by spectrophotometry.
13. Evaluation of composition and stability constant of Fe (III) –Tiron complex by Turner and Anderson method.
14. Assay of asprin/Caffeiene/phenacetin by UV-spectrophotometry.
15. Polarographic determination of Cd and zinc in solutions individually and in a mixture
16. Polarographic evaluation of stability constant of lead oxalate complex.
17. Analysis of mercury/lead in industrial effluents by spectrophotometry.
18. Analysis of DO, BOD and COD of a waste water sample by titrimetry
19. Nephelometric determination of barium and phosphate in domestic waste water.

CA P 558: ANALYTICAL CHEMISTRY PRACTIALS – V

(A minimum of twelve experiments are to be carried out)

1. Determination of ascorbic acid in goose berry/bitter gourd by titrimetry & spectrophotometry.
2. Catalytic determination of iodide/selenium by spectrophotometry.
3. Enzymatic determination of glucose in blood by spectrophotometry.
4. Determination of urinary reducing sugar, chlorides, urea and uric acid by titrimetry
5. Estimation of blood cholesterol by spectrophotometry
6. Determination of blood urea and uric acid by spectrophotometry
7. Assay of urinary creatinine by spectrophotometry.
8. Isolation of Casein and lactose from milk.
9. Isolation of caffeine from tea
10. Preparation of 6-10 Nylon poly (hexamethylene) sebacamide) by interfacial polymerization and its characterization by m.p, inherent viscosity andIR studies.
11. Study of kinetics of polymerization.
12. Determination of molecular weight of polymer by viscosity and turbidimetry.

13. Analysis of phenol-formaldehyde reaction products by TLC

Computer related experiments: The following exercise may be given to illustrate the use of Softwares such as Excel and Origin in calculation and plotting curves using the data generated in regular lab experiments.

1. Use of mathematical functions to calculate parameters such as ionic strength, rate constants, dissociation constants, energy of activation, standard deviation, average molecular weights of polymer samples or any other similar calculation.
2. Use of software to make linear plots and calculate constants from slope and intercept-data from experiments such as verification of beer's law, determination of pKa of weak acids from pH data, determination of energy of activation , viscosity with concentration for determination of unknown concentration/ average molecular weight of polymers or any other similar data sets.
3. Use of software to fit multiple set of data obtained in different series of experiments on the same chart- pka of different weak acids, kinetic data with different ionic strength conditions etc-or any other series of data may be given.
4. Use of software to fit non-linear curves with data from experiments such as absorbance vs. wavelength, first derivative curves of potentiometric and pH titrations, radioactive decay or any other similar experiments .

References:

1. A text book of quantitative Inorganic Analysis – A.I. Vogel
2. Vogel's text book of quantitative Inorganic analysis –Basset, Denney, Jeffery, & Mendham
3. Colorimetric Determination of Traces of Metals – E.B. Sandell
4. Computers in chemistry. K.V.Raman,TMH,1993
5. Computers in Chemistry & Instrumentation,Vol.1-5 Mattson, Marcel Dekker, NY,1974
6. Numerical methods in chemistry, K.J Johnson , Marcel Dekker,NY,1980

CA P 559: PROJECT WORK AND DISSERTAION