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M.Sc\_Analytical Chemistry Practical\_Syllabus.doc

**Adikavi Nannaya University :: Rajahmundry**  
**Department of Chemistry**  
**M.Sc. (Final) Chemistry Syllabus for III-Semester**  
**Specialization: Analytical Chemistry**  
**Paper - I: Separation Methods – I**  
(Effective from 2016-17Admitted batch)

**Unit – I Chromatography - 1**

**Chromatography:** classification of different chromatographic methods, methods of development-Elution development, Gradient elution development, displacement development, and frontal analysis.

Principles of chromatography, different migration, adsorption phenomena, partition, adsorption coefficient, retardation factor, retention time and volume, column capacity, temperature effects, partition isotherm.

Dynamics of chromatography-efficiency of chromatographic column, zone spreading, High Equivalent Theoretical Plate (HETP), Van Deemter equation, resolution, choice of column, length and flow velocity, qualitative and quantitative analysis.

**Unit - II Chromatography – 2**

**Column chromatography (adsorption chromatography):** principles, general aspects, adsorption isotherms, chromatographic media, nature of forces between adsorbent and solutes, eluents (mobile phase), column chromatography without detectors and liquid chromatography with detectors and applications.

**Gel Exclusion chromatography or Gel filtration chromatography:** principles, properties of xerogels, apparatus and detectors, resolution of gel type, applications to organic compounds.

**Capillary Electrophoresis :** Principle, Details of the Instrument, Applications to Inorganic and Organic compounds.

**Unit – III Chromatography – 3**

**Gas chromatography:** Theory, Instrument description of equipment and different parts, columns (packed and capillary columns), detector specifications-thermal conductivity detector, flame ionization detector, electron capture detector, nitrogen-phosphorus detector, photo ionization detector, programmed temperature gas chromatography; applications in the analysis of gases, petroleum products etc., other detectors used their Principles and Applications.

**Inorganic molecular sieves:** structure of zeolites, crystals, types of sieves, application in the separation of gases including hydrocarbons, ion exclusion-principles and applications,

Counter current chromatography-principles and application, Affinity chromatography- principles and applications

### ***GC-MS – Introduction***

Instrumentation – GC – MS interface – Mass spectrometer (MS) Instrument operation, processing GC – MS data – ion chromatogram Library searching – Quantitative measurement – sample preparation Selected ion monitoring – Application of GC-MS for Trace constituents. Drugs analysis, Environmental analysis and others.

## **Unit – IV Chromatography – 4**

***Liquid-liquid partition chromatography:*** principle, supports, partitioning liquids, eluents, reverse phase chromatography, apparatus, applications

***High performance liquid chromatography:*** Theory, Instrument description of the different parts of the equipment, columns, detectors-UV detector, refractometric detector, Fluorescence detector, Diode Array detector, applications in the separation of organic compounds, names of other detectors used their Principles and Applications.

***LC-MS*** – Introduction – Instrumentation – liquid chromatograph – Mass spectrometer Interface – Instrumental details – Processing LC-MS data – ion chromatograms – Library searching – Quantitative measurements. Sample preparation – selected ion monitoring. Application of LC-MS for Drug analysis, Environmental samples and others.

### **Text books:**

1. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
2. M.N. Sastri, Separation methods, Himalaya Publishing Company, Mumbai

### **Reference books:**

1. E. Helfman, Chromatography, Van Nostrand, Reinhold, New York
2. E. Lederer and M. Lederer, Chromatography, Elsevier, Amsterdam.
3. Chemical separation methods, John A Dean, Von Nostrand Reinhold, New York
4. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
5. H.M Mc Nair and J. M. Miller, Basic Gas Chromatography, John Wiley, New York
6. W. Jeumings, Analytical Gas chromatography, Academic Press, New York
7. H. Eugelhardt (ed), Practice of HPLC, Springer Verrag, Berrin

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**Specialization: Analytical Chemistry**  
**Paper- II: Quality control and Traditional methods of Analysis-I**  
(Effective from 2016-17 Admitted batch)

**Unit – I: Quality control in Analytical Chemistry**

- (a) *Characteristics of an analysis:* quality of an analytical procedure, limit of detection, sensitivity, safety, cost measurability, selectivity and specificity, quality control-principles of Ruggedness test, control charts, Youden plot, and ranking test.
- (b) *Evaluation and reliability of analytical data:* limitation of analytical methods, accuracy, precision, errors in chemical analysis, classification of errors, minimization of errors, significant figures, computations and propagation of errors.
- (c) *Statistical analysis:* Mean deviation, Standard deviation, coefficient of variance, normal distribution, F test, T test, rejection of results, presentation of data.
- (d) *Quality assurance and management systems:* elements of quality assurance, quality assurance in design, development, production and services, quality and quantity management system, **ISO 9000** and **ISO 14000** series-meaning of quality, quality process model, customer requirement of quality calibration and testing, statistical process control, process control tools, control chart, statistical quality control, acceptance sampling.  
Good laboratory practices (GLP) – need for GLP, GLP implementation and organization, GLP status in India.
- (e) Brief out line of ICH guide lines on drug substances and products.

**Unit – II Decomposition techniques in analysis**

**(a) Inorganic Compounds**

Principle of decomposition and Dissolution. Difference between dissolution / decomposition of Organic and Inorganic substances.

Importance of Decomposition Techniques in Analysis.

Principle of Dissolution of an inorganic substance.

Decomposition of samples with acids – H<sub>2</sub>O, HCl, HF, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub> and HClO<sub>4</sub>

Decomposition of samples by fusion, Principle and with two examples each

Alkali Fusion--- Na<sub>2</sub>CO<sub>3</sub>, NaOH,

Acidic Fusion--- Sodium Hydro Sulphate, Sodium Pyro Sulphate

Oxidation Fusion---Na<sub>2</sub>O<sub>2</sub>, Sodium Chlorate

Reductive Fusion Na<sub>2</sub>CO<sub>3</sub> + Na<sub>4</sub>BO<sub>4</sub>

What is Sintering process, How is it different from Fusion.

Fusion with alkali carbonates, alkali hydroxides, Sodium Peroxide

Decomposition of samples by sintering with sodium peroxide, sodium carbonate.

Principles of decomposition at high temperatures, high pressures .

Principles of Microwave and ultrasonic decomposition techniques.

**(b) Organic Compounds**

Principles of solubility of organic compounds, non polar, polar solvents.  
Recrystallisation methods and application of solubility and Recrystallisation.

**Unit – III Oxidant systems – Principles and applications in analysis**

Analytical chemistry of some selected oxidant systems – formal, standard and normal potentials in various media, species responsible for the oxidation properties, stability of the solutions, standardization, requirement for the selections of the oxidants, selection of suitable indicators for Oxidant systems.

- a) Inorganic Systems Mn (III), Mn (VII), Ce (IV), Cr (VI), V (V), periodate, iodate,
- b) Organic Systems chloramine-T.

**Unit – IV Organic Functional group analysis**

Classification of functional groups with suitable examples.

Determination of:

- 1) Functional groups imparting acidic nature – thiol, enediol, phenolic hydroxyl.
- 2) Functional groups imparting basic nature – Aliphatic and Aromatic primary, secondary and tertiary amines – hydrazine derivatives.
- 3) Functional groups which impart neither acidic nor basic nature – Aldehydes, Ketones, Nitro, Methoxy, Olifinic.

**Text books:**

- 1. Technical methods of analysis – Griffin, Mc Graw Hill Book Co.
- 2. Chemical Separation and measurements – D.G Peterseti, John M.Haves Sanders Co.
- 3. Chemical analysis – H.A Laitinan, Mc Graw Hill Book Co.
- 4. Newer redox titrants – Berka, Zyka and Vulterin, Pergamon Press
- 5. Volumetric Analysis, Vol III – I.M Kolthoff and R. Belcher, Interscience Public, New York
- 6. Vogel's Text Book of Inorganic Quantitative Analysis – J. Bassett et al, ELBS
- 7. Organic functional groups – S. Siggia

**Reference Books:**

- 1. D.A Skoog, D.M West and F.J Holler, Analytical Chemistry, An Introduction, Sanders College Publishing, New York
- 2. K.V.S.G Murali Krishna, An Introduction ISO 9000, ISO 1400 Series, Environmental Management
- 3. Quality Assurance and Good Laboratory Practices, Prof. Y. Anjaneyulu, In Now Publication, New York
- 4. Quality Assurance in Analytical Chemistry – G.Kateman and F.W Pijpers, John Wiley and Sons, New York
- 5. Quantitative Chemical Analysis – I.M Kolthoff, E.B Sandel, E.J Meehan, S. Bruckenstein, Macmillan Company, London
- 6. Decomposition Techniques in Inorganic Analysis – J.Dolezal, P.Povondra, Z.Sulcek

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**Paper – III: Applied Analysis-I**  
(Effective from 2016-17 Admitted batch)

**Unit – I Analysis of Ores**

- (a) General techniques of analysis applied to complex materials - Scope of metallurgical analysis -  
General methods of dissolution of complex materials - Various chemical methods for the effective separation of the constituents in the complex materials.
- (b) Analysis of ores: Iron ore- Analysis of the Constituents – Moisture , loss of ignition, Total Iron, ferrous Iron ,Ferric Iron, alumina , silica, Titania, Lime, Magnesia, Sulphur, phosphorous, manganese, alkalies, combined water, Carbon in blast furnace, flue dust and sinter.
- (c) Manganese Ore - Analysis of the Constituents – Total Manganese,  $\text{MnO}_2$ ,  $\text{SiO}_2$ ,  $\text{BaO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ , P and S
- (d) Chromite Ore - Analysis of the Constituents – Chromium,  $\text{SiO}_2$ ,  $\text{FeO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ , &  $\text{MgO}$ .
- (e) Phosphate rock Ore - Analysis of the Constituents -  $\text{CaO}$ ,  $\text{P}_2\text{O}_5$ , F,  $\text{SiO}_2$ ,  $\text{CO}_2$ , S,  $\text{Na}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , MgO,  $\text{K}_2\text{O}$ , Cl, MnO. Organic carbon, Moisture, Loss of ignition.
- (f) Aluminium Ore (Bauxite) - Analysis of the Constituents – Silica, Alumina,  $\text{Fe}_2\text{O}_3$ , Titania, MnO,  $\text{P}_2\text{O}_5$ ,  $\text{CaO}$ , MgO, vanadium, zirconium, and alkalies.

**Unit – II Analysis of Finished Products – I**

- (a) Analysis of steel for C, Si, S, P, Mn, Ni, Cr; Mg and analysis of blast furnace slag .
- (b) Analysis of refractory materials: fire clay, flour spar, and magnesite
- (c) Analysis of fluxes - limestone and dolomite.

**Unit – III Analysis of Finished Products – II**

- (a) Chemical Analysis of cement-silica,  $\text{NH}_4\text{OH}$  group, ferric oxide, alumina, lime, magnesia, Sulphide Sulphur ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ , free  $\text{CaO}$  in Cement and Clinker,  $\text{SO}_3$  and loss on ignition.
- (b) Analysis of oils - saponification number, iodine number, and acid number..
- (c) Analysis of soaps - moisture, volatile matter, total alkali, total fatty matter, free caustic alkali or free fatty acids, sodium silicate , chloride.
- (d) Analysis of paints-vehicle and pigment,  $\text{BaSO}_4$ , total lead and lead chromate

## Unit – IV Assessment of water Quality

Sources of water, classification of water for different uses, types of water pollutants and their effects,

Analytical methods for the determination of the following ions in water:

Anions:  $\text{CO}_3^{2-}$ ,  $\text{HCO}_3^-$ ,  $\text{F}^-$ ,  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$ ,  $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{CN}^-$ ,  $\text{S}^{2-}$

Cations:  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{As}^{5+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$

Determination of Dissolved oxygen (D.O), Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD), standards for drinking water.

### Text books

1. Handbook of Analytical Control of Iron and Steel Production, Harrison John, Wiley 1979
2. Standard methods of Chemical Analysis, Welcher
3. Technical Methods of Analysis, Griffin, Mc Graw Hill
4. Commercial Methods of Analysis, Foster Dee Snel and Frank M. Griffin, Mc Graw Hill Book Co.
5. Water Pollution, Lalude, Mc Graw Hill
6. Environmental Chemistry, Anil Kumar De, Wiley Eastern Ltd.
7. Environmental Analysis, S.M. Khopkar (IIT Bombay)

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**Paper – IV: INSTRUMENTAL METHODS OF ANALYSIS - I**  
(Effective from 2016-17 Admitted batch)

**Unit – I : Spectroscopic Methods - 1**

- (a) ***UV-Visible Spectroscopy:*** laws of absorption, deviation from Beer's law, single and double beam spectrophotometers-instrumentation, sources of radiation, detectors, qualitative analysis by absorption measurements, general precautions in colorimetric determinations, determination of certain metal ions by using ligands –  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{NH}_4^+$ ,  $\text{Cr}^{3+}$ ,  $\text{Cr}^{6+}$ ,  $\text{Co}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$  and anions –  $\text{NO}_2^-$ ,  $\text{PO}_4^{3-}$  using suitable reagents, simultaneous determinations of dichromate and permanganate in a mixture, spectrophotometric titrations, principle of diode array spectrophotometers.
- (b) ***Spectrofluorimetry:*** Theory of fluorescence, phosphorescence, factors affecting the above, quenching, relation between intensity of fluorescence and concentration, instrumentation, application with reference to  $\text{Al}^{3+}$ , chromium salts, fluorescence, thiamin (B1) and riboflavin (B2) in drug samples.

**Unit – II : Spectroscopic Methods - 2**

- (a) ***Infrared spectroscopy:*** units of frequency, wavelength and wave number molecular vibrations, factors influencing vibrational frequencies, instrumentation, sampling techniques, detectors, characteristic frequencies of organic molecules, qualitative and quantitative analysis with reference to (petroleum refinery and polymer industry), selected molecules like CO,  $\text{CO}_2$ , non-destructive IR method for the analysis of CO and other organic compounds, principles of Fourier transform IR.
- (b) ***Raman Spectroscopy:*** Raman effect and spectra, differences between Raman spectra and IR spectra, instrumentation, Raman spectra of CO,  $\text{CO}_2$ ,  $\text{N}_2\text{O}$ ,  $\text{H}_2\text{O}$ .

**Unit – III : Spectroscopic Methods -3**

- (a) ***NMR Spectroscopy:*** resonance condition, origin of NMR spectra, instrumentation, chemical shift, factors affecting chemical shift, shielding, spin-spin splitting, mechanism for spin-spin coupling, interpretation of NMR spectra of typical organic compounds, factors influencing NMR spectra, fast chemical reactions, magnitude of I, nuclei with quadrupole moments, FT NMR, study of isotopes other than proton- $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$ ,  $^{11}\text{B}$ , double resonance, spin tickling, shift reagents, applications.



(b) *ESR Spectroscopy*: principle, g value, hyper fine splitting, qualitative analysis, Krammers degeneracy, fine splitting, instrumentation, introduction to double resonance technique, difference between ESR and NMR spectra, quantitative analysis, application to study of free radicals and other analytical applications.

#### **Unit – IV : Spectroscopic Methods -4**

(a) *Mass Spectroscopy*: Principle, basic instrumentation, energetics of ion formation, types of peaks observed, resolution, qualitative analysis, molecular weight determination, quantitative analysis, advantages

(b) *X-ray Spectroscopy (XRF)*: chemical analysis by X-ray spectrometers, energy dispersive and wavelength dispersive techniques, evaluation methods, instrumentation, matrix effects, applications.

#### **Text Books:**

1. Instrumental methods of analysis – H.H Willard, Meritt Jr. and J.A Dean
2. Principles of instrumental analysis – Skoog and West
3. Vogels Textbook of Quantitative Inorganic analysis – J. Basset, R.C Denney, G.H Jefferey and J.Madhan
4. Instrumental methods of analysis – B.K Sarma, Goel Publishing House, Meerut
5. Instrumental methods of Analysis – Chatwal and Anand
6. Instrumental methods of Analysis – Ewing
7. Handbook of ICP
8. The ICP – Bogdain B.

#### **Reference Books:**

1. Applications of ICP-MS, A.R Date and A.L Glay, London (Eds), Blackie, London
2. A. Moutaser and D.W Golightly (Eds), ICP in Analytical Atomic Spectrometry, VeH Publishers, New York
3. G.I Moore, Introduction to ICP emission Spectrometry in Analytical Spectroscopy, Elsevier, Amsterdam

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**Paper-1: Separation Methods – II**  
(Effective from 2016-17 Admitted batch)

**Unit – I Chromatography - 5**

- (a) **Paper chromatography:** principle, papers as a chromatographic medium, modified papers, solvent systems, mechanism of paper chromatography, experimental technique, different development methods-ascending, descending, horizontal, circular spreading, multiple development, two dimensional development, reverse phase paper chromatographic technique-visualization and evaluation of chromatograms, applications.
- (b) **Thin layer chromatography:** principle, chromatographic media-coating materials, applications, activation of adsorbent, sample development, solvent systems, development of chromatoplate, types of development, visualization methods, documentation, applications in the separation, HPTLC-principle, technique, applications.

**Unit – II Chromatography - 6**

- (a) **Ion Exchange:** principles of ion-exchange systems, synthetic ion-exchange resins, properties of anion and cation exchange resins, ion-exchange mechanism, ion-exchange equilibria, selectivity, ion-exchange capacity, applications of ion-exchangers in different fields.
- (b) **Ion exchange chromatography:** Principle, Equipment, Application Specifically Separations of Lanthanides, Actinides, amino acids.
- (c) **Ion chromatography:** principles of separation, instrumentation, detectors, separation of cations and anions, applications in the analysis of water and air pollutants.

**Unit – III Sampling of Solids, Liquids and Gases**

**Sampling:** Basis of sampling, purpose of sampling, homogeneous and heterogeneous samples, statistical criteria for good sampling, sample size, sampling unit, gross sample, laboratory sample.

Sampling of Solids: Cone and Quartering method, Long pile and alternative shovel method, precautions in preservation of solid samples, sampling of metals and other solids rods, wires, sheets, plates, especially Gold, Silver, Iron and other metals.

Sampling of different types of liquids: different sampling techniques, sampling of drinking water, industrial effluents, precautions in sampling and preservation of collected liquid samples.

Sampling of gases: sampling and Preconcentration by adsorption or absorption method, instantaneous monitoring, sampling in samplers and subsequent monitoring, different types of gas samplers, precautions in preservation of samples, systematic sampling and random sampling.

#### **Unit – IV Importance of Analytical chemistry & Solvent Extraction**

**(a) Importance of Analytical Chemistry to Industrial Research:** Importance of Qualitative and Quantitative analysis in research and development, industries and other branches of science.

Development and validation of an analytical method, units, concentrations, calculations, standards, chemical reactions, expressions of concentrations, importance of separation methods with examples.

**(b) Solvent Extraction:** principles and processes of solvent extraction, Distribution Law and Partition coefficient, nature of partition forces, different types of solvent extraction systems – Batch extraction, Continuous extraction, Counter current extraction, solvent extraction systems, applications in metallurgy, general applications in analysis and pre-concentration, special extraction systems like crown ethers, super fluid and surfactant extractions-examples.

#### **Text books:**

1. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
2. Separation methods, M.N Sastri, Himalaya Publishing Company, Mumbai

#### **Reference books:**

1. E. Helfman, Chromatography, Van Nostrand, Reinhold, New York
2. E. Lederer and M. Lederer, Chromatography, Elsevier, Amsterdam.
3. Chemical separation methods, John A Dean, Von Nostrand Reinhold, New York
4. R.P.W Scott, Techniques and practice of Chromatography, Marel Dekker Inc., New York
5. E.Stahl, Thin layer chromatography, Academic Press, New York
6. James, G.Tartor (Ion chromatography)

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**Paper – II : Traditional Methods of Analysis - II**  
(Effective from 2016-17 Admitted batch)

**Unit – I Precipitation methods - 1**

- (a) Crystal habit and super saturation, nucleation and crystal growth, homogeneous and heterogeneous nucleation, solubility and particle size, colloids, completeness of precipitation, effect of excess precipitant, pH, complex formation, temperature, purity of precipitates, aging.
- (b) **Co-precipitation and post precipitation** : theory of adsorption of salts having an ion in common with the main precipitate, co-precipitation in colloidal precipitates, adsorption of solvents, mixed crystal formation by occlusion and entrapment, re-precipitation with examples, Post-precipitation – theory of post-precipitation, examples of post-precipitation, conditions for obtaining pure and quantitative precipitates.
- (c) **Precipitation Titrations**: Principle, Indicators for precipitation titrations, determination of halides.

**Unit – II Precipitation methods - 2**

- (a) **Precipitation from Homogeneous Solution (PFHS)**: theory of PFHS, methods of PFHS – increase in pH, decrease in pH, cation release, anion release, reagent synthesis, change in oxidation state, photochemical reactions, precipitation from mixed solvents. Applications of PFHS methods.
- (b) **Gravimetric determinations**: nature of species, preparation of solutions, limitations, interferences, inorganic precipitants-chloride and sulphate, organic precipitants dimethyl glyoxime (DMG), oxine, benzidine, salicylaldehyde, benzoin oxime, sodium tetraphenyl boron, tetraphenyl arsonium chloride.
- (a) **Electro-gravimetric analysis**: principle, important terms in electrogravimetry, decomposition voltage or decomposition potential, over voltage and their importance, instrumentation, electrolysis at constant current, determination of  $\text{Cu}^{2+}$  by constant current electrolysis, electrolysis at controlled potentials, determination of Cu, Pb, Sn in brass and bronze by controlled potential electrolysis.

**Unit – III Reductant system – Principles and applications in analysis**

Analytical chemistry of some selected reductant systems – formal, standard and normal potentials in various media, stability of the solutions, species responsible for

the reduction properties, standardization, requirement for the selection of the reductants, selection of suitable indicators for various reductant systems,

- (a) Inorganic Systems – Cr (II), V (II), Ti (III), Sn (II), Fe (II) in  $\text{H}_3\text{PO}_4$  and hydrazine,
- (b) Organic Systems – hydroquinone and Ascorbic acid.

#### **Unit – IV Analysis of some selected Drugs:**

Basic considerations of drugs – Classification

Determination of the following Drugs:

- 1) Acetyl salicylic acid ( Antipyretic – Analgesic )
- 2) Testosterone, progesterone and cortisone (Steroids and corticoids)
- 3) Sulphadiazine ( sulphadugs)
- 4) Phenobarbitone (Barbituric acid derivatives)
- 5) Chloramphenicol, Benzyl penicillin and Tetracycline (Antibiotics)
- 6) Thiamine (B1), Riboflavin (B2) and ascorbic acid (c) [Vitamins]
- 7) Isoniazid ( Antimicrobial agents)
- 8) Methyldopa (Antihypertensive agents)
- 9) Metronidazole (Antiamoebic agents).

#### **Text books:**

- 1. Technical methods of analysis – Griffin, Mc Graw Hill Book Co.
- 2. Chemical Separation and measurements – D.G Peterseti, John M.Haves Sanders Co.
- 3. Chemical analysis – H.A Laitinan, Mc Graw Hill Book Co.
- 4. Newer redox titrants – Berka, Zyka and Vulterin, Pergamon Press
- 5. Volumetric Analysis, Vol III – I.M Kolthoff and R.Belvher, Interscience Public, New York
- 6. Vogel's Text Book of norganic Quantitative Analysis – J.Bassett et al, ELBS
- 7. Pharmaceutical analysis – T. Higuchi, Brochmann hausfen

#### **Reference Books:**

- 1. D.A Skoog, D.M West and F.J Holler, Analytical Chemistry, An Introduction, Sanders College Publishing, New York
- 2. Quantitative Chemical Analysis – I.M Kolthoff, E.B Sandel, E.J Meehan, S. Bruckenstein, Macmillan Company, London

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**Paper – III: Applied Analysis – II**  
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**Unit – I Analysis of raw materials**

- (a) Analysis of non-ferrous alloys:
- (i) Brass – Analysis of the constituents – Cu, Zn, Sn, Pb and Fe.
  - (ii) Bronze - Analysis of the constituents – Cu, Sn, Zn, Pb and Fe.
  - (iii) Solder - Analysis of the constituents – Sn, Pb and Sb.
- (b) Analysis of Ferro alloys :
- (i) Ferro silicon - Analysis of the constituents – Si, C, P, S
  - (ii) Ferro vanadium - Analysis of the constituents – V, C, P, S, Si, Al.
  - (iii) Ferro manganese - Analysis of the constituents – Mn, S, C, P, Si
  - (iv) Silico manganese - Analysis of the constituents – Mn, S, C, P, Si
  - (v) Ferro chromium - Analysis of the constituents – Cr, C, Si.

**Unit – II Analysis of Soil, Fertilizer and Fuel**

- (a) Analysis of soils: sampling, determination of moisture, total N, P, Si, lime, humus nitrogen, alkali salts, soil absorption ratio.
- (b) Analysis of fertilizers: ammonical fertilizers, Phosphate fertilizers, Nitrate fertilizers.
- (c) Analysis of fuels: solid fuels-coal, proximate analysis, ultimate analysis, heating value, grading of coal based on Ultimate Heat Value(UHV).

**Unit – III Assessment of Air Quality**

Composition of pure air, classification of air pollutants, toxic elements present in dust and their sources – collection of air samples.

Sources, effects, control of pollution and chemical analysis for the following.

- (a) Primary pollutants:
- (i) Carbon compounds - Carbon monoxide(CO) and Carbon dioxide(CO<sub>2</sub>).
  - (ii) Sulphur compounds- sulphur dioxide (SO<sub>2</sub>), Sulphur trioxide (SO<sub>3</sub>) and Hydrogen Sulphide (H<sub>2</sub>S).
  - (iii) Nitrogen compounds - nitric oxide (NO), and nitrogen dioxide (NO<sub>2</sub>),
  - (iv) Hydrocarbons - Aliphatic hydrocarbons and polycyclic aromatic hydrocarbons (PAH).
  - (v) Particulate matter - Respirable and Suspended particulate matter, Inorganic and Organic particulates.

- (b) Secondary pollutants - ozone ( $O_3$ ), peroxy acetyl nitrate (PAN), peroxy benzyl nitrate (PBN)
- (c) Standards for ambient air quality.

#### **Unit- IV Kinetic Methods of Analysis & Non aqueous Titrimetry**

- (a) Kinetic methods of analysis: introduction, slow reactions, catalyzed reactions, methods of determination of catalyst concentration, extrapolation method for the determination of catalyst, variable time method, fixed time method, examples for the determination of toxic metals and anions using some typical kinetic reactions.
- (b) Non aqueous titrimetry : Classification of solvents and titrations for non aqueous titrimetry- Types of reactions - Indicators .
- (i) Determination of acids
  - (ii) Determination of bases
  - (iii) Karl-Fisher reagent for the determination of moisture content in drugs and other samples.

#### **Text books**

1. Chemical analysis – H.A Laitinan, Mc Graw Hill Book Co
2. Standard methods of Chemical Analysis, Welcher
2. Technical Methods of Analysis, Griffin, Mc Graw Hill
3. Commercial Methods of Analysis, Foster Dee Sneel and Frank M. Griffin, Mc Graw Hill Book Co.
4. Environmental Chemistry, Anil Kumar De, Wiley Eastern Ltd.
5. Environmental Analysis, S.M Khopkar (IIT Bombay)
6. Environmental Air Analysis, Trivedi and Kudesia, Akashdeep Pub.

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**Paper - IV: Instrumental Methods of Analysis -II**  
(Effective from 2016-17 Admitted batch)

**Unit – I: Spectro-analytical Methods of Analysis**

(a) *Flame photometry*: theory, instrumentation, combustion flames, detectors, and analysis of Na, K, Ca, Mg

(b) *Atomic Absorption Spectrometer*: theory, instrumentation, flame and non-flame techniques, resonance line sources, hollow cathode lamp, instrumentation, chemical and spectral interferences, applications with special reference to analysis of trace metals in oils, alloys and toxic metals in drinking water and effluents

(c) *Inductively coupled plasma spectrometer (ICP-AES, ICP-MS)*: principles, instrumentation, plasma, AES detectors, quadrupole mass spectrometers, difference between the two detectors, analysis methods for liquids and solids, applications in the analysis of trace and toxic metals in water, geological and industrial samples.

(d) *Arc and Spark spectrographic Direct analysis of solid for metals.*

**Unit – II Thermal methods of Analysis**

(a) Thermo gravimetry-theory, instrumentation, applications with special reference to  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{CaC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ,  $\text{CaCO}_3$ ,  $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$

(b) Differential thermal analysis-principle, instrumentation, difference between TG and DTA - applications with special reference to the clays and minerals, coals (fuels)

(c) Differential scanning calorimetry-principle, instrumentation, applications to inorganic materials like chlorates and per chlorates, ammonium nitrate, organic compounds and Drugs.

**Unit- III : Electro analytical Methods of Analysis - 1**

(a) *Voltametry and polarographic analysis* : principle of polarography, residual current, migration current, diffusion current, half-wave potential, Ilkovic equation, instrumentation, Dropping mercury electrode (DME), advantages and disadvantages of DME, qualitative and quantitative analysis of inorganic ions-Cu, Bi, Pb, Cd, Zn, AC polarography, pulse polarography

(b) *Anode stripping voltametry*: principle, instrumentation, Hanging mercury drop electrode, application in the analysis of Pb and Cd in environmental samples, principle of cathode stripping voltametry.



- (c) **Coulometric analysis:** principles of coulometric analysis with constant current, coulometric analysis with controlled potential, applications of coulometric methods for the analysis of cations-As (III), Fe (II) and I<sup>-</sup> and S<sup>2-</sup> by using I<sub>2</sub> liberations and Ce<sup>4+</sup> liberation in solutions

#### **Unit – IV Electro Analytical and Radio chemical methods of analysis - 2**

- (a) **Ion Selective Electrodes:** reference electrodes - hydrogen electrode, calomel electrode, silver chloride electrode; indicator electrodes – hydrogen and glass electrodes, theory of membrane potentials and liquid junction potentials, types of ion selective electrodes, basic properties, potentials and construction, calibration of ion selective electrodes, ion selective electrodes with fixed membrane sites, silver, lead, cadmium, sulfide, fluoride, cyanide and glass electrodes, applications in the analysis of air and water pollutants, principles of liquid membrane, gas sensing and enzyme based electrode
- (b) **Radio chemical methods of analysis:** detection and measurement of radioactivity, introduction to radioactive tracers, applications of tracer technique, isotope dilution analysis - applications, activation analysis – application, advantages and disadvantages, radio carbon dating technique

#### **Text Books:**

1. Instrumental methods of analysis – H.H Willard, Meritt Jr. and J.A Dean
2. Principles of instrumental analysis – Skoog and West
1. Vogels Textbook of Quantitative Inorganic analysis – J. Basset, R.C Denney, G.H Jefferey and J.Madhan
2. Instrumental methods of analysis – B.K Sarma, Goel Publishing House, Meerut
3. Instrumental methods of Analysis – Chatwal and Anand
4. Instrumental methods of Analysis – Ewing

#### **Reference Books:**

W.Wendtlandt, Thermal Analysis, John Wiley Sons, New York

**Adikavi Nannaya University :: Rajahmundry**  
**Department of Chemistry**  
**M.Sc. (Final) Chemistry Syllabus for III-Semester**  
**Specialization: *Analytical Chemistry***  
**ANALYTICAL CHEMISTRY PRACTICAL – I**  
(With effect from 2016-2017 admitted batch)

**Classical Methods of Analysis-1**

1. Water analysis
  - (i) Determination of total hardness ( $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ ) of water samples
  - (ii) Determination of chloride ( $\text{Cl}^-$ ) present in water samples
  - (iii) Determination of dissolved oxygen (DO) of drinking water and sewage water
  
2. Complexometric titrations
  - (i) Determination of the concentration of calcium in milk powder by complexometric titration (EDTA)
  - (ii) Determination of Calcium and Magnesium in limestone or dolomite samples using EDTA.
  
3. Fertilizer analysis
  - (i) Determination of ammonia from ammonia containing fertilizer
  - (ii) Determination of phosphate from fertilizer
  
4. Analysis of iron ore
  - (i) Complete analysis of iron ore
  - (ii) Determination of percentages of Fe (II) and Fe (III) present in iron ore sample
  
5. Analysis of Coal
  - (i) Determination of moisture content of coal sample
  - (ii) Determination of volatile matter of coal sample
  - (iii) Determination of fixed carbon of coal sample
  - (iv) Determination of ash content of coal sample

## **Instrumental Methods of Analysis-1**

1. pH metry
  - (i) Determination of alkalinity of a coloured effluent using pH metric titration.
  - (ii) Determination of purity of commercial HCl using pH metric titration.
  - (iii) Determination of purity of commercial H<sub>2</sub>SO<sub>4</sub> using pH metric titration.
  
2. Potentiometry
  - (i) Determination of Cr(VI) with Fe(II) using potentiometric end point
  - (ii) Determination of Fe (II) using ceric sulphate by potentiometric end point
  - (iii) Determination of a mixture of Ce(IV) and V(V) with Fe(II) by potentiometric end point
  - (iv) Determination of KSCN with AgNO<sub>3</sub> by potentiometric end point.
  
3. Spectrophotometry
  - (i) Determination of Fe (III) using potassium thiocyanate
  - (ii) Determination of Iron(II) using orthophenanthroline
  - (iii) Determination of phosphate in fertilizer and cola drinks by Molybdenum blue method
  - (iv) Determination of Manganese (II) -periodate method
  
4. Flame photometry
  - (i) Determination of sodium present in bread samples
  - (ii) Determination of sodium and potassium in a given sample of fertilizer
  
5. Thin layer chromatography: Determination of R<sub>f</sub> values and identification of organic compounds in a given mixture by TLC
  - (i) Separation of mixture of benzil and 2-nitrophenol
  - (ii) Mixture of benzophenone and naphthalene
  - (iii) Mixture of 2-nitrophenol and 4-nitrophenol

### References:

1. A Text Book of Quantitative Inorganic Analysis (3<sup>rd</sup> Edition) – A. I. Vogel

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**ANALYTICAL CHEMISTRY PRACTICAL – II**

(With effect from 2016-2017 admitted batch)

**Classical Methods of Analysis-2**

1. Water analysis
  - (i) Determination of alkalinity ( $\text{CO}_3^{2-}$ ,  $\text{HCO}_3^-$ ) of water samples.
  - (ii) Determination of chemical oxygen demand (COD) of drinking water and sewage water
  - (iii) Determination of biological oxygen demand (BOD) of drinking water and sewage water
  
2. Redox titrations
  - (i) Determination of oxalate in kidney stones by permanganometric titration.
  - (ii) Determination of Fe(II) present in an Iron tablet using  $\text{KMnO}_4$
  
3. Fertilizer analysis
  - (i) Determination of nitrate from fertilizer
  - (ii) Determination of sulfur (as sulfate) from sulfur containing fertilizer.
  
4. Analysis of oils and soaps
  - (i) Determination of saponification value, acid value and iodine value of oil sample
  - (ii) Determination of moisture content and total alkali of soaps
  
5. Separation and determination of ions by ion-exchanger resins
  - (i) Determination of  $\text{Na}^+$  by cation exchanger resin
  - (ii) Determination of  $\text{Na}^+$  and  $\text{K}^+$  in a mixture by cation exchanger resin
  - (iii) Determination of  $\text{Cl}^-$  and  $\text{Br}^-$  in a mixture by anion exchanger resin

## Instrumental Methods of Analysis-2

### 1. pH metry

- (i) Determination of purity of commercial  $\text{H}_3\text{PO}_4$  by pH metric titration
- (ii) Determination of  $\text{CH}_3\text{COOH}$  by pH metric titration.
- (iii) Determination of stability constant of copper glycinate

### 2. Potentiometry

- (i) Determination of Fe(II) using Mn(VII) of by potentiometric titration
- (ii) Determination of Fe (II) using V(V) of by potentiometric titration
- (iii) Determination of a mixture of Mn(VII) and V(V) with Fe(II) using potentiometric end point
- (iv) Determination of a mixture of bromide and chloride with  $\text{AgNO}_3$  using potentiometric end point

### 3. Spectrophotometry

- (i) Determination of nitrite in drinking water samples by diazotization method
- (ii) Determination of nitrate -phenoldisulphonic acid method
- (iii) Simultaneous Determination of Cr(VI) and Mn(VII) in a mixture without separation
- (iv) Determination of Cu(II) using EDTA – Photometric titration method.

### 4. Flame photometry

- (i) Determination of Lithium by flame photometry
- (ii) Determination of calcium from milk samples using flame photometry

### 5. Thin layer chromatography

- (i) Separation and identification of the given mixture of colourless compounds (Diphenylamine, Benzophenone and Naphthalene)
- (ii) Separation and identification of the given mixture of coloured compounds (azobenzene, hydroxyazobenzene, p-aminoazobenzene).

### References:

1. A Text Book of Quantitative Inorganic Analysis (3<sup>rd</sup> Edition) – A. I. Vogel