

# **SOLAPUR UNIVERSITY, SOLAPUR**

## **SYLLABUS OF M. Sc. Part-II (Analytical Chemistry)**

A two-year duration M.Sc. Analytical Chemistry syllabus is prepared by a panel of eminent University professors, scientists and experts from industries. It is designed for the Semester system and this syllabus will be implemented with effect from June 2013 for M.Sc. II after approved by appropriate academic bodies and the University authorities. The syllabus has been prepared taking into consideration the present and near future Needs; SET, NET, UGC guidelines and syllabi of other established Universities.

### **General Structure of the Course:**

The course consists of four semesters spread over two academic years. Each semester has four "theory papers" of 100 marks each (including 30 marks of internal tests). For semester I and II each all four papers are compulsory and for semesters III and IV three papers are compulsory and fourth is elective. There are four practicals, each I and II semester, where as practicals of M. Sc. II are, spread over Sem.III & IV. Examinations of these practicals will be held of the end of each semester. The distribution of marks is mentioned below.

M Sc Part-I

Theory Paper (Semester I and II):  $8 \times 100 = 800$  marks (including 30 marks of internal tests)

Practical (Semester I and II)  $4 \times 50 \times 2 = 400$  marks

M Sc Part-II

Theory Paper (Semester III and IV):  $8 \times 100 = 800$  marks (including 30 marks of internal tests)

Practical (Semester III and IV) = 400 marks

Total: 2400 marks

Ratio of marks of Theory: Practical:  $(1600:800) = 2:1$

## **M. Sc. Part-I (Analytical Chemistry)**

Syllabus for M Sc Part-I (Analytical Chemistry) is same as M. Sc. Part-I (Organic /Polymer /Industrial /Physical Chemistry) course of the university.

## **M. Sc. Part-II (Analytical Chemistry) (From June 2013)**

### **Semester III**

**CH-ANAL-301 : Advance Separation Techniques**

**CH-ANAL-302 : Instrumental Methods of Analysis-I.**

**CH-ANAL-303 : Applied Analytical Chemistry.**

### **Optional Papers**

**CH-ANAL-304-A : Analytical Spectroscopy.**

**CH-ANAL-304-B : Analysis of Commercial Materials.**

### Practicals

**CH-ANAL-305 : Practical-V**

**CH-ANAL-306 : Practical-VI**

## Semester IV

**CH-ANAL-401 : Advance Analytical Techniques**

**CH-ANAL-402 : Instrumental Methods of Analysis-II.**

**CH-ANAL-403 : Bio-Chemical and Food Analysis.**

### Optional Papers

**CH-ANAL-404-A : Environmental Chemical Analysis.**

**CH-ANAL-404-B : Pharmaceutical Analysis.**

### Practicals

**CH-ANAL-405 : Practical-VII**

**CH-ANAL-406 : Practical-VIII**

### NATURE OF PRACTICAL EXAMINATION.

M. Sc. Part-II: Distribution of marks (Practicals):-

Sem. III:	<b>305 (V)</b>	<b>306 (VI)</b>
1. Experiments	60	60
2. Internal Practical Exam.	30	30
3. Oral examination	05	05
4. Journal	05	05
<b>Total</b>	<b>100</b>	<b>100</b>

\* Practical Exam will be conducted for 3days; with 2 experiments each day. (I.e. total 6 Experiments-for 60 + 60 = 120 marks-)

Sem. IV:	<b>405 (VII)</b>	<b>406 (VIII)</b>
1. a) Experiments	70	--
b) Lab Project / Review Article-literature / Industrial Training	--	50
2. Internal Practical examination	30	30
3. Oral examination	--	10 (of Expts)
4. Journal	--	10
<b>Total</b>	<b>100</b>	<b>100</b>

\*\* Practical Exam will be conducted for 3days; with 2 experiments each day for 2 days. (I.e. total 4 experiments-for 70 marks) and on one day project-defence should be carried out.

### Nature of Question Papers

There shall be seven questions out of which the candidate has to solve five questions. Each question will carry 14 marks. Q. NO.1 (Section I) will be compulsory, objective type and will carry 1 mark per sub-question. Out of Q. No. 2 to 4 (Section II) and Q. No. 5 to 7 (Section III) candidate has to solve two questions from each section, total four questions. At least 50% of the questions should be problem oriented rather than descriptive, keeping in view SET, NET, GATE and other competitive examinations. The questions should test the understanding of the candidate rather than memory. The purpose of the exercise should be career oriented to prepare the candidate well for national and international level competition.

**Notes:**

1. M.Sc. I syllabus is common for Polymer Chemistry, Industrial Chemistry, Organic Chemistry, Physical Chemistry, Analytical Chemistry and Inorganic Chemistry.
2. It is absolutely essential to have yearly inspection of the Laboratories of the affiliated Colleges [where M. Sc (Chemistry) is being taught] by the 3 member Committee appointed by the Solapur University.
3. Each theory course prescribed for M. Sc. should be covered in 48 periods, each of 60 minutes; engaging 4 lectures per course per week. (Including tutorials, seminars etc.)
4. Each practical course will require 6 hr of Laboratory work per week. [i.e. 12 hr for 2 Lab. Courses per week]
5. There shall be 8 students in a batch for M. Sc. Practical Course. (In any case not more than 10 students in a batch).

**CH-ANAL-301****Advance Separation Techniques-I****Unit-I****12**

**Paper Chromatography**-Definition, theory and principle, techniques, one, two dimensional and circular paper chromatography, mechanism of separation, structure of cellulose and types of paper, methodology-separation of sample, choice of solvents, location of spots and measurement of  $R_f$  value, factors affecting  $R_f$  value, advantages and applications.

**Affinity Chromatography**- Introduction, theory stationary phase, preparation of column, separation of antigens.

**Exclusion Chromatography**- Theory and principle of size exclusion chromatography, experimental techniques for gel filtration chromatography (GFC) and gel-permeation chromatography (GPC), materials for packing-factors governing column efficiency, methodology and applications.

**Unit-II****12**

**Electrophoresis**-Theory and classification, factors affecting mobility, macromolecular size and charge interactions with supporting electrolyte, pH and concentration discontinuities, Factors affecting electrophoresis phenomena-electrolysis, electrosomosis, temperature and supporting media. Instrumentation, methodology, Preparation of gelstaining and destaining, preparative zone electrophoresis, continuous electrophoresis and Applications.

**Capillary Electrophoresis**-Principle, theory, instrumentation, sample preparation and applications, Capillary electrochromatography and Miscellar electrokinetic capillary chromatography

**Unit-III****12**

**Membrane-Based Methods:-Dialysis**-working of techniques, membranes, general consideration of diffusion, Donnan Membrane equilibrium and Applications.

**Electrodialysis**- working of techniques, membranes, Electrodialysis cells and Applications.

**Ultrafiltration**- working of techniques, membranes, non-gelatinous membranes and Applications. Dialysis compared with other membrane-separation methods.

**Other Separation Methods:-Ultracentrifugation**-Principle, sedimentation constant, sedimentation equilibrium, sedimentation velocity, methodology and applications.

**Zone refining-** Principle, zone leveling and applications.

#### **Unit-IV**

**12**

**Solvent Extraction-** Basic principles, classification of solvent extraction systems, Factors affecting extraction process, Mechanism of extraction, extraction by chelation extraction by solvation, extraction equilibria for chelates, extraction equilibria for solvation, techniques of extraction, separation of metals by extraction, extraction by ion-pair formation, application of solvent extraction in industry, solid-phase extraction (SPE) and uses.

#### **References**

1. Solvent Extraction in Analytical Chemistry. By G H Morrison and H Freiser ( John Wiley New York, 1958)
2. Solvent Extraction of metals by A K Da, S M Khopkar and R A Clalmers. (Von Nonstrant Ravinhold, 1970)
3. A Textbook of Inorganic Qualitative Analysis. By A I Vogel.
4. Chromatography. By E Heftman, 5<sup>th</sup> edition, part-A and part-B, Elsewhere Science Publisher, 1992.
5. Chromatography Today. By C F Poole and S K Poole, Elsewhere Science Publisher, 1991.
6. Electrophoresis- Analytical Chemistry. Open Learning by M Melvin John Wiley and Sons.1987.
7. Analytical Chemistry. By G D Christian 4<sup>th</sup> edition, John Wiley and Sons, 1986.
8. Instrumental Methods of Analysis, by B K Sharma, 19<sup>th</sup> edition, Goel Pubisher,2000.
9. Basic Concept in Analytical chemistry, by S.M. Khopkar.

### **CH-ANAL-302**

#### **Instrumental Methods of Chemical Analysis-I**

##### **Unit-I**

**12**

**Thermal Methods of Analysis-**Introduction, thermo gravimetric analysis (TGA),types of thermo gravimetric analysis, principle, factors affecting the results-heating rate, furnace, instrument control/data. Application-purity and thermal stability, evaluation of correct drying temperature, analysis of complex mixture and determination and kinetic parameters of thermal degradation.

**Differential Thermal Analysis(DTA)-** Theory, variables affecting the DTA curves, Difference between TGA and DTA. General principle, instrumentation, applications. Analysis of physical mixtures and thermal behaviors study. Determination of melting point, boiling point and decomposition point.

**Differential Scanning Calorimetry (DSC)-**Basic principal, difference between DTA and DSC. Instrumentation-Power compressed DSC, Heat flux DSC. Applications- Studies of thermal transition and isothermal crystallization. Thermometric titrations.

##### **Unit-II**

**12**

**Radio Analytical Techniques-**Determination of nuclear radiation and counting devices. Radioactivity tracers- Principal and applications. Isotopic Analysis- direct and inverse.

Special analytical application-radiometric titrations. Neutron activation analysis: principle, instrumentation, applications and limitations. Radiochromatography and Radioimmunoassay: principle, instrumentation, applications and limitations.  
Radiometric titration- principle, types, instrumentation and applications

### **Unit-III**

**12**

#### **Electro Analytical Techniques**

**1. Ion Selective Electrodes-** Terminology, types and construction of selective electrodes glass electrode, solid state and precipitate electrodes, liquid-liquid membrane electrode, enzyme and glass-sensing electrodes, glass electrodes for pH measurement (glass micro electrode) and applications

**2. Voltametry and Stripping voltametry-** Principle, instrumentation and applications. Cyclic voltametry.

Electrogravimetric analysis- Theory, apparatus, cell process, deposition and separation, electrolytic separation of metals and applications.

#### **3. Coulometric Methods**

Introduction, Principle, Coulometric techniques, potentiostatic coulometry, Coulometric titrations, Coulometric determination, Advantages, Instrumentation for coulometric titration and applications.

### **Unit-IV**

**12**

**1. High frequency titration** principle, instrumentation, advantages and disadvantages of high frequency method. Applications acid-base, complexometric measurement dielectric constant and analysis of mixture.

**2. Amperometry titration** principles of Amperometric titrations, instrumentation, titration procedure, advantages and disadvantages

**3. Electrogravimetric titration** principle & theory, methods of electrogravimetric analysis, instrumentation, advantages and disadvantages

### **References**

1. Thermal methods of Analysis by W W Wendlandt.
2. Radioactivity applied to chemistry. By Arthur C Wahs and R J Words
3. An introduction to radiation chemistry. By J W T Spinks and R J Words.
4. Fundamentals of analytical chemistry. By D A Skooj, D M West, Holler and Crowh. 5<sup>th</sup> edition. Sounders College publishing New York.
5. Analytical Chemistry. By G D Christian 5<sup>th</sup> edition 2005. John Wiley and Sons Inc. India.
6. Vogel's Textbook of Quantitative Chemical Analysis. By J Mendham R C, Denney J D, Barnes and MJK Thomas. 6<sup>th</sup> edition. Third Indian Reprint 2003. Pearson Education. Pvt. Ltd. New Delhi.
7. Radioisotopes Techniques MGH by Overman and Clark
8. Radio-bioassay by Faund S. Ashkar, Volume-I page 1-35 and 53 to 65 CRC press, Inc. Boca Ratin, Florida.
9. Basic Concept in Analytical chemistry, by S.M. Khopkar.
- 8 Instrumental methods of chemical analysis, by H. Kaur (Pragati Prakashan,)

## CH-ANAL-303

### Applied Analytical Chemistry.

#### Unit-I

12

##### Agricultural Analysis-I

**Soil Analysis-** Soil sampling, field description of soils, physical analysis, determination of major and minor constituents, exchange capacity, soil reaction (pH), chemical analysis as a measure of soil fertility.

**Stock feeds analysis** -feeding stuffs, qualitative analysis and quantitative analysis.

**Plant analysis-**Preparation of sample, moisture determination- methods of ashing, methods of plant analysis- starch, sugars, determination of mineral constituents (Fe, Mn, Mo, Si, Ca, Mg, P, S, C and N).

#### Unit-II

12

##### Agricultural Analysis-II

**Analysis of Fertilizers** - Sampling, sample preparation. Analysis of nitrogen, phosphorus and potassium. Nitrogen: urea nitrogen, Kjeldahl nitrogen method, Ammonia nitrogen, phosphorus: total phosphorus. Alkalimetric ammonium molybdophosphate method, potassium : potassium by sodium tetraphenyl borate method.

**Pesticides and insecticides analysis-**Introduction, classification, analysis of DDT, gammexane, , zinab, ziram, thiram, thiometon, and chloridane.

#### Unit-III

12

##### Analysis of Minerals, Ores and Alloys

**Minerals and ores-** Hematite, pyrolusite, gypsum, dolomite, bauxite, and illmenite ores.

**Metal and alloys analysis-** Steel, Cu-Ni alloy, solder, bronze, brass, aluminium alloy, Chromium.

#### Unit-IV

12

##### Cosmetics Analysis

Composition of creams and lotions, determination of water, propylene glycol, non-volatile matter and ash content. Determination of borate, sulphate, phosphate, chlorides, titanium and zinc oxide.

##### Analysis of face powder

Estimation of boric acid, magnesium, calcium, zinc, iron, aluminium and Barium

##### Analysis of Deodorants and antiperspirants

Composition, analysis of fats and fatty acids, boric acid, Mg, Ca, Zn, Fe, Ti, Al, phenol, hexachlorophenone, methanamine, sulphonate and urea.

#### References

1. Agricultural Analysis. By Kanwar.
2. Soil Analysis. By Jackson.
3. Encyclopedia of Industrial Methods of Chemical Analysis. By F D Snell ( All senus)
4. Quantitative Inorganic Analysis. By A I Vogel.
5. Standard Methods of Chemical Analysis. By F J Welchar.
6. Instrumental methods of chemical analysis by Chatwal and Anand.

7. Instrumental methods of chemical analysis by Merit, Dean, Settle
8. Analytical Instrumentation, Handbook, Marcel Dekker Inc. (1987) by Garen U.Ewing,
9. Principle and practice of Analytical chemistry by F.U.Fifield and D.Keuley 3<sup>rd</sup> edition,Blackie and sons Ltd.
10. Cosmetics By W. D. Poucher
11. Instrumental methods of chemical analysis H. Kaur

## CH-ANAL-304-A

### Analytical Spectroscopy

#### Unit-I 12

**Raman spectroscopy**-Introduction, principle, quantum mechanical theory of Raman effect, classical theories of Raman effect, rotational Raman spectra, vibrational Raman spectra, Vibrational-rotational spectra, Instrumentation, Applications of Raman Spectroscopy.

**Resonance Raman Spectroscopy** : Introduction, technique, difference between normal and resonance Raman spectrum, Resonance Raman effect, Applications of resonance Raman spectroscopy .

#### Unit-II 12

**Mossbauer spectroscopy**- Theory, isomer shift and quadrupole interaction, spectra of iron and tin compounds. Mossbauer spectrophotometer, applications. [ $\text{Fe}_3(\text{CO})_{12}$ , Prussian blue, oxyhemecrythrin, tin halides, hexacyano ferrates, nitroprussides]

**Nuclear Quadrapole Resonance Spectroscopy**- Introduction, effect of magnetic field on the spectra, relation between electric field gradient and structure applications of NQR.

#### Unit-III 12

**Photoelectron Spectroscopy**-Basic, principle of ESCA instrumentation, scope and limitations of ESCA. Ultraviolet photoelectron spectroscopy, Comparison between ESCA and UPS spectroscopy.

**Photo acoustic spectroscopy**-Basic principles of photo acoustic spectroscopy (PAS), PAS gases and condensed system, chemical and surface applications.

#### Unit-IV 12

##### Electron Spin Resonance Spectroscopy

Electron behaviour, ESR spectrometer, spectra, hyperfine interaction, free radical and interpretation of the spectra. Applications in quantitative analysis. Numerical problems.

**Electron Microscopy**-classification of electron microscopy methods, scanning electron microscopy, working of SEM instrument, comparison of probe microscopy (SPM or STM). Scanning tunnelling microscopy (STM)-basic principles, atomic force microscopy (AFM), application AFM, comparison of electron microscopy with electron spectroscopy.

#### References

1. Fundamentals of Molecular Spectroscopy. By C N Banwell.
2. Electron Spin Resonance. By Assculiein.
3. Molecular Spectroscopy. By G M Barrow
4. Molecular Spectroscopy. By I N Levine. Wiley Interscience.
5. Basic Concept in Analytical chemistry, by S.M. Khopkar.
6. Spectroscopy (Atomic and Molecular) by G R Chatwal and S K Anand
7. Instrumental methods of chemical analysis by H. Kaur

## CH-ANAL-304-B

### Analysis of Commercial Materials.

#### Unit-I

12

**Analysis of petroleum and petroleum products** -Introduction, constituents and petroleum fractionation, quality control, requirements, safety and hazardous aspects, Analysis of petroleum products- specific gravity, viscosity, Doctor test, sulphuric acid absorption, aniline point, vapour pressure and colour determination, cloud point, pour point. Determination of water, neutralization value (acid and base numbers), ash content, sulphur and mercaptan sulphur. Determination of lead in petroleum.

#### Unit-II

12

**Analysis of coal and coke**-Types, composition, preparation of sample, proximate and ultimate analysis calorific value by bomb Calorimetry.

**Analysis of gaseous fuels**-composition of fuel gases, collection of gas, analysis of fuel gases (coal gas, producer gas, water gas and flue gas)

**Analysis of Explosive**-General methods- Heat of explosion, Hygroscopicity, moisture by Karl Fischer titration, Qualitative tests of explosives, Qualitative analysis of explosive mixtures Dynamites. Blasting caps and electric detonators, primers, liquid propellants and solid propellants.

#### Unit-III

12

**Cement** - Loss on ignition, insoluble residue, total silica, sesquioxides, lime, magnesia, ferric oxide, sulphuric anhydride, air and dust pollution from cement plants, atmospheric dispersion of pollutants in cement industry.

**Glass and Glass-Ceramics** - Introduction, composition, method of analysis-sampling and sampling preparation, composition analysis-preliminary testing, decomposition, chemical method for the individual constituents- Si, B, Pb, Zn, Al, Cl, Ca, Mg, Ti.

#### Unit-IV

12

**Analysis of Paints and Pigments**-Preliminary inspection of sample. Test on the total coating. Separation of pigments, binder and thinner of latex paints. Separation of pigments, binder and thinner of solvent type coating. Modification of binder. Identification and analysis of thinner.

**Analysis of Soaps**-method of analysis-sampling, separation, identification, determination of soap composition-fatty acids, total anhydrous soap and combined alkali, potassium, water, determination of inorganic fillers and soap builders, determination of other additives.

**Analysis of Detergents**-types, method of analysis-sampling, separation, identification of components, determination of surfactants and other constituents.

#### References

1. Standard Methods of Chemical Analysis. 6th Ed. Vol I & II (D.Van.Nostard comp) by F.J.Welcher
2. Treatise on Analytical Chemistry Vol.I & II by L.M.Kolthoff
3. Encyclopedia of Industrial chemical Analysis Vol I to 20 (John Wiley) Riech:
4. Outline of Industrial Chemistry by F.D.Snell :
5. Instrumental Methods of Analysis (CBS) by Willard, Merrit, Dean & Settle :

6. Cosmetics by W D Poucher (Three volumes)
7. Handbook of Industrial Chemistry. By Davis Burner.
8. Instrumental Methods of Analysis (CBS) by Willard, Merrit, Dean & Settle
9. Separation Chemistry in Chemistry and Biochemistry. By Roy Keller, M Decker Inc.
10. Quantitative Analysis by Vogel

## **CH-ANAL-401**

### **Advanced Analytical Techniques**

#### **Unit-I**

**12**

##### **Ion Chromatography**

Principle, structure and characteristics of resins, eluent, suppressor columns and detectors used in ion chromatography, analytical applications, environmental speciation by ion chromatography and applications

#### **Unit-II**

**12**

##### **Hyphenated Techniques:-**

Advanced techniques of analysis: - UV-Visible, IR, <sup>1</sup>H-NMR (Recapitulation), <sup>13</sup>CNMR, Mass spectrometry (advanced examples), GC-MS, HPLC-MS and their applications.

**Kinetic methods of analysis**-Rates of chemical reactions, rate laws, first order second order kinetics, pseudo first order and second order reactions, enzyme catalysed reactions, steady state conditions, determination of reaction rates-experimental techniques, types of kinetic methods differential and integral, applications.

#### **Unit-III**

**12**

##### **Automated Analysis**

Automated Systems- An overview, distinction between automatic and automated systems, advantages and disadvantages of automation, types of automated techniques. Non-discrete techniques, segmented flow methods and basic equipments, special techniques and devices, theoretical considerations and problems, applications. Single and multi channel auto analyzers, BUN analyzers, automatic glucose analyzer and ammonia in water analyzer, COD analyzer. Non-segmented flow methods. Flow injection analysis- Principle, types of dispersion, factors affecting dispersion, applications of small, medium and large dispersion, stopped flow methods, flow injection titrations. Discrete methods- Centrifugal fast scan analyzer, automatic multipurpose analyzer, automatic elemental analyzer, automated analyzer based on multilayer film principles, film structure, instrumentation applications. .

#### **Unit-IV**

**12**

**Super critical fluid chromatography (SFC)**-Introduction, properties of super critical fluids, supercritical fluid chromatography-instrumentation and operating variables, comparison to other types of chromatography, applications,

**Super critical fluid extraction (SFE)**- Introduction, advantages of SFE, instrumentation, supercritical fluid choice, Off-line and On-line extraction and applications.

**Particle size determination**-introduction, Low-angle laser light scattering instrumentation, theoretical models and application. Dynamic light scattering-principles, instrumentation and applications. Photosedimentation- settling velocity and particle size, instrumentation and applications.

## References

1. A Textbook of Inorganic Qualitative Analysis. By A I Vogel.
2. Chromatography. By E Heftman, 5<sup>th</sup> edition, part-A and part-B, Elsewhere Science Publisher, 1992.
3. Chromatography Today. By C F Poole and S K Poole, Elsewhere Science Publisher, 1991.
4. Analytical Chemistry. By G D Christian 4<sup>th</sup> edition, John Wiley and Sons, 1986.
5. Instrumental Methods of Analysis, by B K Sharma, 19<sup>th</sup> edition, Goel Pubisher, 2000.
6. Absorption Spectroscopy of Organic Molecules (Addition Wesley) by V.M. Parikh
7. Instrumental Methods of Analysis (CBS) by Willard, Merrit, Dean & Settle :
8. Spectroscopic Methods in Organic Chemistry (Mc Graw Hill) by D.H. Williams & J.Fleming :
9. Silverstein Spectroscopic Identification of Organic Compounds. (John.Wiley)
10. Applications of NMR Spectroscopy of Organic Chemistry (Pergamon Press) by Jackmann & Sternhill :
11. Nuclear Magnetic Resonance (Mc Graw Hill) by J.D.Roberts :
12. Mass Spectroscopy by K.Benjamin :
13. Introduction to Instrumental Analysis by R.D.Brown, Mc.Graw Hill (1987)
14. Instrumental Methods of Chemical Analysis by Chatwal and Anand.
15. Basic Concept in Analytical chemistry, by S.M. Khopkar.

## CH-ANAL-402

### Instrumental Methods of Analysis-II.

#### Unit-I

12

##### X-ray methods of Analysis

Introduction , theory of X-ray generation, comparison with optical spectroscopy, X-ray spectroscopy, X-ray absorption, X-ray diffraction and X-ray fluorescence methods, theory, instrumentation, applications.

**Surface characterization by spectroscopy**-Introduction to the study of solid surfaces, types of surface measurements spectroscopic surface methods, general techniques in surface spectroscopy, sampling of surfaces, surface contamination.

#### Unit-II

12

**Nephelometry and Turbidometry**-Introduction, theory, comparison of spectrophotometry, turbidimetry and nepelometry, instrumentation and applications.

**Refractometry** -Principle, parameters influencing refraction, significance of critical angle during measurements, refractometers, qualitative and quantitative analysis and analytical applications.

**Interferometry**-principles, instrumentation and applications.

#### Unit-III

12

**Fluorimetry and Phosphorimetry**- Introduction, comparison of absorption and fluorescence methods, theory, instrumentation, applications of fluorimetry and phosphorimetry, comparison of fluorimetry and phosphorimetry, comparison of fluorimetry and phosphorimetry with absorption methods, applications of Photoluminescence methods, Chemiluminescence-phenomenon, measurement and applications.

## Unit-IV

12

**A) Emission Spectroscopy**-Introduction, theory, types of emission spectra, excitation energy requirements, relation of emission spectroscopy to flame photometry, instrumentation-excitation sources, electrodes, sample holder, monochromators, detectors, spectrographs, quantitative and qualitative and specific applications, advantages and disadvantages of emission spectroscopy.

**B) Flame photometry** principle-Instrumentation, interferences in flame photometry. Factors that influence intensity of emitted radiations of flame photometry, limitations of flame photometry and applications

### References

- 1) Instrumental Methods of Analysis by U. H. Willard L. L. Merrit, J. A. Dean
- 2) Basic Concept in Analytical chemistry, by S.M. Khopkar.
- 3) Principles of Instrumental Analysis, Holl Seamlers by D. Sleool and U. West
- 4) Kinetics in Analytical Chemistry Inter science NY by H. Mark and G. Rachnitz
- 5) Instrumental Methods of Chemical Analysis, Pragati Prakashan Meerut by H. Kaou,
- 6) A Text book of Quantitative Analysis by A.I. Vogel
- 7) Instrumental methods of chemical analysis by H. Kaur

## CH-ANAL-403

### Bio-chemical and food analysis

#### Unit-I

12

##### Food Analysis

Food flavors, food colour, food preservatives, milk and milk products, flour starches, honey, jam and their major component analysis.

Introduction to natural fats and oils, analysis of oils and fats : softening point, congeal point, titre point, cloud point, Iodine value, saponification value, acid value by R-M and Polenske value, Elaiden test.

#### Unit-II

12

##### Body Fluid Analysis

Composition and detection of abnormal level of certain constituents leading to diagnosis of diseases. Sample collection and preservation of physiological fluids, analytical methods to the constituents of physiological fluids (blood, urine and serum).

Blood-Estimation of glucose, cholesterol, urea, hemoglobin and bilirubin

Urine- urea, uric acid, creatinine, calcium, phosphate, sodium, potassium and chloride.

#### Unit-III

12

##### Drug Analysis

Introduction, source of drug, difference between drug and medicine, dangerous drugs, narcotics, classification of drugs, impurities / contaminants in pharmaceutical raw materials / chemical and drug, drug screening using gas chromatography, assay of drugs.

Characterization and analysis of some of the following drug-molecules;

1. Local anesthetics: Procaine Hydrochloride
2. Sedative-Hypnotics: Phenobarbital
3. Antianxiety agents: Diazepam
4. Anticonvulsants: Phenytoin

5. Antipsychotic agents: Chlorpromazine
6. Central intraneural blocking agents: Levodopa
7. Antidepressant agents: Imipramine hydrochloride
8. Central nervous system stimulants: Fenfluramine hydrochloride
9. Opioid Analgesics and Antitussives: Morphine sulphate, Codeine phosphate
10. Diuretic agents: Hydrochlorothiazide
11. Antihistamines- Antiallergic agents: Mepyramine maleate, Cyclizine hydrochloride
12. Nonsteroidal anti-inflammatory drugs: Ibuprofen, Paracetamol
13. Drugs that affect the cardiac functions: Isosorbide dinitrate, Digitoxin
14. Adrenergic agents: Ephedrine, Adrenaline
15. Antihypertensive agents: Propranolol hydrochloride
16. Cholinergic agents: Pilocarpine nitrate, Neostigmine bromide
17. Barbiturates: Phenobarbital, Cyclobarbitone calcium

#### Unit-IV

12

**Clinical Analysis-** Biological significance, analysis of assay of enzymes (pepsin, monoamine, oxidase, tyrosinase), vitamins(thiamine, ascorbic acid, Vit. A) and hormones (progesterone, oxytocin, insulin) chemical, instrumental and biological assay wherever applicable.

**Forensic Analysis-** Special features of forensic analysis, sampling, sample storage, sample dissolution, classification of poisons, lethal dose, significance of LD-50 and LC-50. general discussion of poisons with special reference to mode of action of cyanide, organophosphate and snake venom. Estimation of poisonous materials such as lead, mercury and arsenic in biological samples,

#### References

1. Hand Book of Sugars (A VI) by S.R. Junk and H.M.Pancoast
2. Perfumery Technology (JC1) by B. Bilal and B.V.Well
3. Treatise on Analytical Chemistry Vol I and Vol II
4. Laboratory Techniques in Food Analysis by I.M.Kolthof, D.Pearson
5. Handbook of Analysis and Quality, Control for Fruits and Vegetable Products 2<sup>nd</sup> Ed Mc.Graw hill) by S.Ranganna
6. Aids to the Analysis of Food and Drug by Nicholls
7. Standard Methods of Chemical Analysis vol I and II (6<sup>th</sup> ed) by F.J.Welcher
8. Analysis of Food Products. (Swan Publishers) by S.N.Mahendur :
9. Chemical Analysis of food by Pearson.
10. Textbook of Forensic Pharmacy by B M Mithal 9<sup>th</sup> edition 1993, National Centre Kolcutta.
11. Practical Pharmaceutical Chemistry by Beckett.
12. Textbook of Forensic pharmacy- B. M. Mithal 9<sup>th</sup> Edn (1993) National Centre, Calcutta.
13. Forensic Pharmacy by B.S Kuchekar, and A.M Khadare Nirali Prakshan)
14. Analytical Chemistry by Alka Gupta Pragati Prakashan
15. P.D.Sethi, Quantitative analysis of drugs in pharmaceutical formulations, Unique publisher, New Delhi.
16. S.N.Pandeya, A Text Book of Medicinal Chemistry Vol.I and Vol.II, S.G. Publisher, Varanasi.
17. Alka L.Gupta, Analytical chemistry, Pragati prakashan, Merut.

## CH-404-A

### Environmental Chemical Analysis

#### Unit-I

12

**Environment**-Concept of biosphere and its components: Hydrosphere, atmosphere and lithosphere. Hydrological cycles. Formation of soil, Bio-geochemical cycles, C, N, P, S and O. Bio distribution of elements.

**Air pollution**- Suspended particulate matter, aerosol. Generation, hazards and control of SO<sub>x</sub>, NO<sub>x</sub> and CO<sub>x</sub>. Monitoring equipment – cyclone separator, electrostatic precipitator, filters and settling chambers.

#### Unit-II

12

##### Water-

Water resources, origin of waste water, types of water pollutants and their sources.

Chemical analysis of water: Objectives of analysis, parameters of analysis, sample collection and preservation, measurement of colour, turbidity, total solids, alkalinity, hardness, chloride, residual chlorine, sulphates, fluorides, phosphates and different forms of nitrogen in natural and polluted water and heavy metals. Pollution control. Public health significance of Pb, Cd, Hg, As, Cu, Zn and Mn.

Pollution control measures.

General survey of instrumental techniques for the analysis of heavy metal in aquatic system.

Organic loadings-significance and measurement of DO, BOD, COD, TOD and TOC, phenols, pesticides, surfactants, and tannin and lignin as water pollutants and their determination.

#### Unit-III

12

**Waste water treatment**- Terminology in waste water treatment, waste water characteristics, effluent standards.

Treatment of domestic waste water: **a) Preliminary treatment.**

**b) Primary treatment**-sedimentation, equalization, neutralization.

**c) Secondary treatment**-aerated lagoons, trickling filters, activated sludge process, oxidation ditch, oxidation pond and anaerobic digestion, sludge treatment and disposal.

**d) Tertiary treatment**-evaporation, ion-exchange, adsorption, electro dialysis, electrolytic recovery and reverse osmosis.

**e) Advanced waste water treatment**-nutrient removal-nitrogen and phosphorus removal, solids removal.

**f) Waste water disposal and re use, industrial waste water and its treatment**

#### Unit-IV

12

**Soil Pollution**-Source of soil pollution, soil sediments as a pollutants, treatment and remedial measurements of soil pollution. Soil loss and degradation.

**Industrial pollution**- Sugar industry, paper and pulp industry, nuclear power plant, polymer drugs, radionuclide analysis, disposal of wastes and their management.

Principles of decomposition -biodegradability, better industrial process.

**Hazardous substance analysis**- Nature, Source, Physical classification of hazardous substances Chemical classification hazardous wastes, Physical and chemical methods of waste treatment and ultimate disposal of hazardous waste.

Case studies-Bhopal gas tragedy, Chernobyl nuclear reactor accident, Three mile island tragedy, Minamata disaster.

## References

- 1) Environmental Chemistry, Environmental Pollution Analysis by S.M.Khopkar,
- 2) Environmental Chemistry, New Age International Publisher by A.K.De
- 3) Industrial Chemistry by B.K.Sharma
- 4) Introduction to Instrumental Analysis by R.E.Brown, Mc.Graw Hill (1987)
- 5) Fundamentals of Analytical Chemistry 6<sup>th</sup> edition by D.A.Skoog, D.M.West and F.S.Holler.
- 6) Instrumental Methods of Chemical Analysis by Chatwal and Anand.
- 7) Water and Waste Water Analysis; (NEERI) Ramteke,
- 8) Industrial Chemistry by B.K.Sharma.
- 9) Pollution Control in Process Industries by S.P.Mahajan.
- 10) Measurements of Air Pollutants. By M Katz, WHO, 1969.
- 11) Handbook of Air Pollution. By Stern, APHA , 1980.

## CH-ANAL-404-B

### Pharmaceutical Analysis.

#### Unit-I

12

##### a) Sources of Impurities in Pharmaceutical Raw Materials and Finished Products:

Raw materials, Method of manufacture, Contamination-atmospheric, particulate, cross contamination, microbiological, process errors, Packing errors, chemical instability, container contamination (in brief) physical changes, temperature effects. General manufacturing processes.

b) Stability Studies, Shelf Life Fixation for Formulated Products

#### Unit-II

12

##### Test and assay of raw materials and finished products :

a) Biological Assays

Introduction, Precision of biological assays in brief, (estimation of errors is excluded) Biological assay of insulin, Tetanus antitoxin, Determination of proteolytic activity, Determination of ABO group and Rh group, Photometric haemoglobinometry, Haemolysins?

b) Chemical Tests and Assays:

Limit test, characteristics of limit tests, specificity sensitivity, control of personal errors, Loss on drying ( NaCl), loss on ignition ( ZnO), limit test for lead, arsenic, chloride and sulphate, moisture determination of moisture by KFR titration method and assay of steroids. steroids (IP)

c) Analysis of vegetable drugs :

Sampling, foreign organic matter, ash values and water soluble ash (ginger) Acid insoluble ash, sulphated ash.

d) Microbiological tests and assays

Microbiological assay of antibiotics, (std. preparations and units of activity, test organisms and inoculum, Apparatus, Method -Cylinder or cup plate method and two level factorial assay ( ampicillin from table 4 I.P.) Microbial limit test {preliminary testing, Mediumsoyabean casein digest agar medium only)and total microbial count only } Test of sterility-Membrane filtration method , Determination of Thiomersal

e) Physical tests and assays

Disintegration tests for (tablets, capsules, pessaries and suppositories Dissolution testtablets and capsules.

### Unit-III

12

#### **Standardization and quality control of different dosage form**

Brief introduction to different dosage forms with the IP requirements Analytical methods for the following- Tablets (aspirin) additives used in tablet manufacture, 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 reporting protocols.

### Unit-IV

12

#### **Role of FDA in pharmaceutical industry.**

Drug cosmetic act Definitions Drug, Misbranded , Adulterated and Spurious drug, New drug Cosmetics ,Blood bank, Manner of labeling, GMP in brief (Schedule M), FDA. Role of FDA, introduction to new drugs, brief summary of different phases of test and approval for formulation of a drug.

#### **References:-**

- 1) Practical Pharmaceutical chemistry third edition volume 1. By A.H.Beckett & J.B.Stenlake
- 2) Pharmacopoeia of India Volume I and II.
- 3) Remington's Pharmaceutical sciences.
- 4) Forensic pharmacy by B.S Kuchekar, A.M Khadare (Nirali Prakashan)
- 5) Practical pharmaceutical analysis by Ashitosh Kaur
- 6) Analytical problems of drug substances and Exp by Florey
- 7) The theory and practice of Ind pharmacy Leon lachmann, Herbert Liebermann and Joseph L.Karnic 3rd edition By Varghese Publication House, Hind Rajasthan Building Dadar Mumbai –14

### CH-ANAL-305: Practical-V

- 1) Estimation of COD from waste water.
- 2) Analysis of waste water with respect to (a) alkalinity (b) T.D.S. (c) Sulphate (d) dissolved chlorine.
- 3) Estimation of nitrogen from given fertilizer by Kjeldahl method.
- 4) Estimation of phosphorus from given fertilizer by volumetry/ colourimetry.
- 5) Estimation of potassium from given fertilizer by gravimetry / Flame photometry.
- 6) Estimation of calcium in the sample of dairy whitener by flame photometry.
- 7) Estimation of sodium in the sample of dairy whitener by flame photometry.
- 8) Analysis of borax by conductometry.
- 9) Determination of iron in syndets by colorimetric method.
- 10) Determination of alcohol from beverages spectrophotometrically using dichromate.
- 11) Dissolution test of tablets.
- 12) Determination of amount of Zinc from the given sample solution by Nephelometric/Turbidimetric titration using standard solution of Ba (NO<sub>3</sub>)<sub>2</sub> or Pb (NO<sub>3</sub>)<sub>2</sub>.
- 13) Determination of amount of chloride from the given sample solution by Nephelometric/Turbidimetric titration using standard solution of AgNO<sub>3</sub>.
- 14) Determination of dissociation constant of weak acid pH-metrically.

- 15) Chromatographic separation of sugars, amino acids by paper, TLC and ion exchange method (both quantitative and qualitative method) separation of organic compounds by column chromatography.
- 16) Estimation of Al in steel Rhudrescence method.
- 17) Analysis of felspar ore.
- 18) Analysis of some common pesticides insecticides, plastics and detergents.
- 19) Assay of sulphha drugs.
- 20) Chemical analysis of chilli-powder.
- 21) Any other relevant expt. may be added

## CH-ANAL-306: Practical-VI

- 1) To determine the amount of each para nitro-phenol and meta nitro-phenol from the given mixture by spectrophotometric titration using standard solution of NaOH (max-280 nm)
- 2) To determine the amout of each copper and bismuth or copper and iron (III) from the given mixture at 745 nm by spectrophotometric titration using solution of EDTA.
- 3) Determination of relative strength of acetic acid, chloroacetic acid and trichloroacetic acid through mesuring their Ka-value by conductivity measurement methode.
- 4) Determination of the pka value of chloroacetic acid and trichloro acetic acid by potentiometry / pH. metry using standard solution of Na OH.
- 5) Determination of strength of acetic acid from the commercial vinegar sample by potentiometric titration and its confirmation by conductmetric / pH-metric titration using standard solution of NaOH
- 6) Flame photometric estimation of each Na, K, Li and Ca from the given sample mixture by calibration curve method
- 7) Estimation of Zn and Cd from unknown solution by polarographic technique.
- 8) Determination of moisture content in food sample by Karl Fisher reagents.
- 9) Flame photometric estimation of Na/k/Li/Ca from their given sample by working curve method and its conformation by standard addition method.
- 10) Estimation of Na and K / Ca and K from the binary mixture by internal standard method using lithium as internal standard on flame photometry.
- 11) Determination of concentration of sulphuric acid, acetic acis and copper sulphate from their mixture by conductometric titration with std. NaOH.
- 12) Estimation of various transition elements like Zn/Ni/Co/Cd/Al from various commercial samples by complexometric titrations on potentiometer by using mercury electrode
- 13) Determination of dissociation constant of Cu-ammonia complex potentiometrically.
- 14) Determination of an indicator constant and isobestic point of an indicator spectrophotophometrically.
- 15) Simultaneous estimation of Cl and I by potentiometrically.
- 16) Estimation of bicarbonate and carbonate by potentiometrically.
- 17) Estimation of purity of a given azo dye by colorometry
- 18) Analysis of iodized table salt.
- 19) Analysis of malathion by colorometry.
- 20) Determination of concentration of given solution by standard addition method.
- 21) Any other relevant expt. may be added

## CH-ANAL-405: Practical -VII

- 1) Estimation of Tannin from Tea.
- 2) Isolation of caffeine from tea.
- 3) Assay of soaps and detergent.
- 4) Preparation of sulphanylde from acetanilide, (Organic Vogel)
- 5) Preparation of methyl Salicylate and assay its purity (O.V)
- 6) Preparation, purification and assay of aspirin.
- 7) Isolation of casein from milk.
- 8) Estimation of the purity of a given azo dye by colourimetry.
- 9) Analysis of milk.
- 10) Determination of sap value and Iodine value of an oil.
- 11) Kjeldahl method for protein estimation in foods and feeds.
- 12) Identification of organic compounds by their IR spectra.
- 13) Determination of amount of vit-B2 in the medicinal tablet fluorometrically.
- 14) Estimation of N-methyl and C-methyl groups.
- 15) Estimation of sodium benzoate/sodium metabisulphite. boric acid and salicylic acid in food.
- 16) Determination of Latent heat of fusion of naphthalene.
- 17) Preparation of sulphanylde from acetanilide (Vogel)
- 18) Preparation of methyl salicylate and assay its purity. (Vogel)
- 19) Estimation of vit.C by 2,6 dichloro-indophenol method.
- 20) Determination of glucose from blood serum.
- 21) Estimation of urea from blood sample.(kit method)
- 22) Any other relevant expt. may be added

## CH-ANAL-406: Practical-VIII

- 1) Determination of amount of zinc from given sample solution by Nephelometric / Turbidimetric titration using standard solution of  $K_4(Fe(CN)_6)$  in 0.4 M HCL
- 2) Determination of amount of sulphate from the given sample solution by Nephelometric / Turbidimetric titration using standard solution of  $Ba(NO_3)_2$  or  $Pb(NO_3)_2$
- 3) Analysis of soda ash.
- 4) Determination of moisture content in food sample by Karl Fisher reagent.
- 5) Spectrophotometric determination of manganese in steel.
- 6) Colorimetric and spectrophotometric determination of manganese in steel.
- 7) Solvent extraction separation of iron from manganese using diethyl ether and their determination titrimetrically.
- 8) Analysis of chrome steel alloy for chromium and nickel content.
- 9) Agricultural analysis of soil sample, animal feeds, soil micronutrients, milk powder for Ca, Fe and P content.
- 10) Simultaneous spectrophotometric determination of (Cr and Mn) and (Ti and V)
- 11) Analysis of Ilmenite ore.
- 12) Estimation of Fe by ceric sulphate and potassium dichromate titration potentiometrically.
- 13) Determination of concentration of Fe ions in ferric salicylate complex spectrophotometrically.
- 14) Estimation of amount of copper (II) with EDTA spectrophotometrically.
- 15) Analysis of vitamin A in food products.

16) Determination of Al/Mg by using 8-hydroxyquinoline as complexing agent by spectrophotometric method.

17) Analysis of pigments with respect to Zn and Cr.

18) Assay of polymer and plastic.

19) Isolation of lycopene from tomato or  $\beta$ -carotene from carrots.

20) Preparation, purification and assay of aspirin.

21) Any other relevant expt. may be added

**Project or Industrial in plant training or Literature Review Articles**

In the final semester, students have to carry out project either at college laboratory or University laboratory or in any recognised R & D laboratory (Public /, Private / Government) or Industry or Institute of national repute across the country under the guidance of a scientist or a post-graduate faculty member. Alternatively, student can undertake literature Review studies. Every student has to submit his / her work in the form of a dissertation; which will be valued by the Examiners during practical examination of Semester IV on the presentation by student.