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Objectives and outcome of the course Analytical Chemistry

Analytical Chemistry is an applied, experimental field of science and is based not only on chemistry, but also on physics, biology, information theory and many fields of technology. It is of fundamental importance not only to all branches of chemistry but also to all biological sciences, engineering sciences, health, medicine, pharmaceuticals, environment, industrial processes, quality control and implementation of legislation.

The objective of B.Sc Analytical chemistry course is to provide students exposure to chemistry, physics, biological sciences, environmental science, computer application, instrumentation and analytical techniques. In this three year course spread over six semesters, there are 10 papers of Analytical chemistry 7 papers of chemistry and 7 Mathematics. In the last semester of this course, there is a provision for one cluster elective papers out of two cluster elective papers, viz.

After graduating in Analytical Chemistry the students can pursue academics in Chemistry, bioinformatics, forensic science, biochemistry and other disciplines of interdisciplinary sciences. They can also use it as a stepping stone to pharmaceutical industry and for Research and Development in industry.

Course Structure

All theory papers will have 4 hours per week and practicals will have 2 hours per week upto Semester IV (Second year). In final year all theory papers will have 3 hours per week and practicals will have 2 hours per week in Semester V and VI (Final year).

Each Theory Paper shall be of 100 marks and Practical Paper shall be of 50 marks.

Total Number of Papers: 24

Mathematics : 7 Papers
Chemistry : 7 Papers
Analytical Chemistry : 7 Papers + 1 Cluster elective (3Papers) = 10 Papers
SEMESTER - I

Paper I - ANALYTICAL CHEMISTRY-I

BASIC PRINCIPLES & LABORATORY OPERATIONS

UNIT - I

I. BASIC CONCEPTS:
   12hrs
   A. SI Units
      i) Definitions of the Seven Base Units (Mass, Length, Time, Temperature, Amount of substance, Electrical current and Luminous intensity), Derived units, Conversion between units, Significant figures.
   B. Chemical concentrations
      i) Mole, molar mass
      ii) Calculations in grams and moles
      iii) Solutions and their concentrations:
      a) Molar concentration
      b) Analytical molarity
      c) Equilibrium molarity of a particular species
      d) Percent concentration
      e) Parts per million/billion (ppm, ppb)
      f) Volume ratios for dilution processes
      g) p-functions.

UNIT - II

INTRODUCTION TO ANALYTICAL CHEMISTRY AND ANALYTICAL METHODS -I

   12hrs
   i) General steps in chemical analysis
   ii) Introduction to methods of detecting analytes
      Physical, Electromagnetic radiations and Electric charge
   iii) Single pan analytical balance: (operation and theory of the balance, construction details, errors in weighing, care of an analytical balance).

UNIT III

INTRODUCTION TO ANALYTICAL CHEMISTRY AND ANALYTICAL METHODS -II

   12hrs

Description and use of common laboratory apparatus: Volumetric flasks, burettes, pipettes, meniscus readers, weighing bottles, different types of funnels chromatographic columns, chromatographic jars, desiccators, drying ovens, filter crucibles, rubber policeman. Calibration and use of volumetric glass ware. pH meter: components of pH meter, use of pH Meter, maintenance of pH meter, application of data. Laboratory notebook.
UNIT – IV

ERRORS IN CHEMICAL ANALYSIS

Types of errors
Accuracy and Precision, Absolute and relative uncertainty, propagation of uncertainty. The Gaussian distribution, mean and standard deviation, confidence intervals. Statistical tests of data (the F test, the t test, Q test for bad data, the method of least squares). Calibration curve.
Laboratory notebook. Safety with chemicals and waste.

UNIT – V

PRINCIPLES OF THERMOGRAVIMETRY:

Practical-I (At the end of Semester-I)

1. Use and calibration of volumetric equipment (volumetric flasks, pipette’s and burette’s).
2. Preparation of standard solutions of acids and bases.
3. Estimation of sodium carbonate by titrating with hydrochloric acid.
4. Preparation of standard solution of EDTA.
5. Estimation of magnesium using EDTA.
8. Preparation of buffers.

SUGGESTED BOOKS

5. Freifelder and Kealy: Analytical Chemistry.
SEMESTER – II

Paper II - ANALYTICAL CHEMISTRY-2

QUANTITATIVE METHODS OF ANALYSIS

UNIT – I

GRAVIMETRIC ANALYSIS - I

A. Precipitation methods
B. Volatilization methods. (The analyte or its decomposition products are volatilized at a suitable temperature. The volatile product is then collected and weighed, or, alternatively, the mass of the product is determined indirectly from the loss in mass of the sample. E.g., determination of the sodium hydrogen carbonates content of antacid tablets)

UNIT – II

GRAVIMETRIC ANALYSIS - II

A. Properties of precipitates and precipitating reagents: Particle size, Filterability of Precipitates (factors that determine particle size, formation of precipitates and particle size) - Colloidal Precipitates (coagulation of colloids, peptization of colloids, treatment of colloidal precipitates) - Crystalline Precipitates (particle size and filterability) - Co-precipitation (surface adsorption, mixed-crystal formation, occlusion, and mechanical entrapment, co precipitation errors) - Precipitation from Homogeneous Solution (The use of the technique of homogeneous solutions to effect precipitation).
B. Drying and Ignition of precipitates
C. Practical gravimetric procedures.

UNIT – III

VOLUMETRIC ANALYSIS

A. Definitions: Titrimetry, Volumetric titrimetry, Gravimetric titrimetry, Coulometric titrimetry.
B. The equivalence point, the end point
Classification of volumetric methods, theory of indicators and buffers - Equilibria Principles - Aqueous and non-aqueous acid-base titration - Redox titrations - Complexometric titrations - Precipitation titrations
C. Typical problems in volumetric titrimetry:
D. Sigmoidal Titration Curves
E. The Henderson-Hasselbalch Equation.
UNIT – IV
CENTRIFUGATION METHODS:

A. Introduction
B. Sedimentation and relative centrifugal force
C. Different types of rotors. D. Density gradient
E. Types of centrifugation techniques.

UNIT – V
INTRODUCTION TO ENVIRONMENTAL ANALYSIS:

A. Sampling method
B. Environmental pollution from industrial effluents and radiochemical waste.
C. Introduction to water and waste analysis.
LABORATORY COURSE -II

Practical-II  Quantitative Analysis

(At the end of Semester-II)

1. Determination of the pKa and Equivalent Weight of a weak acid by pH titration.
2. Determination of the strength of the given magnesium sulphate solution using EDTA and Eriochrome black T as the indicator.
3. Determination of the capacity of an anionic exchange resin.
4. Separation of cadmium and zinc on an ion exchange resin.
5. Homogeneous precipitation of the Nickel as its Dimethylglyoxime.
6. Analysis of soil
   i) Determination of pH of soil.
   ii) Determination of total soluble salts.
   iii) Determination of carbonate and bicarbonate.
   iv) Determination of calcium, magnesium and iron.

Suggested Readings:

2. Laboratory Handbook of Chromatographic Methods (O. Mikes, R.A. Chalmers).
SEMESTER – III
Paper III - ANALYTICAL CHEMISTRY-3

60hrs (4h/w)

SEPARATION METHODS - I

UNIT – I
SOLVENT EXTRACTION:

Introduction, principle, techniques, factors affecting solvent extraction, Batch extraction, continuous extraction and counter current extraction. Synergism, Application - Determination of Iron (III)
ION EXCHANGE: Introduction, action of ion exchange resins, separation of inorganic mixtures, applications, Solvent extraction: Principle and process

UNIT – II
CHROMATOGRAPHY:

A. Classification of chromatographic methods: Principle of differential migration, description of the chromatographic process, distribution coefficients, modes of chromatography, performing column chromatography.
B. Chromatography – theory and practice: Introduction, the chromatograph (elution time and volume), capacity factor, column efficiency and resolution, sample preparation

UNIT – III

A. Techniques of paper chromatography: experimental modifications, various modes of development, nature of the paper, detection of spots, retardation factors, factors that affect the reproducibility of RF values (due to paper, solvent system, sample, development procedure), selection of solvent, quantitative analysis, applications
B. Thin layer chromatography: stationary phase, adsorbents, liquid phase supports, plate preparation, mobile phase, sample application, development, saturation of chamber, detection of spot, RF values (effect of adsorbent, solvent, solute, development process), quantitative analysis, applications

UNIT – IV
COLUMN CHROMATOGRAPHY:

A. General: columns, matrix materials, stationary phase, column packing, application of sample, column development and sample elution, detectors and fraction collectors, applications.
B. High performance liquid chromatography: Principle, column, matrices and stationary phases, column packing, mobile phase and pumps, application of sample, detectors, applications.
C. Adsorption chromatography: Principle, adsorbents, solvents, nature of solute, operating parameters, retention volumes and times, applications.

UNIT – V

B. Ion-exchange chromatography: Principle, ion exchangers, ion-exchange equilibria, ion-exchange resin selectivity, column operations (column development, detection of solute bands), factors affecting retention volumes, applications.

LABORATORY COURSE -III  
Semester-III:  
Practical-III PRACTICAL ANALYTICAL CHEMISTRY  
(At the end of)

30 hrs (2 h / w)

1. Determination of Rf value of amino acids using paper chromatography.
2. Separation and identification of monosaccharide present in a given mixture by paper chromatography.
3. Determination of equivalent conductance of a weak electrolyte (acetic acid) at different concentrations.
   i) Chicory in coffee powder,
   ii) Foreign resin in asafetida
   iii) Chilli powder
   iv) Turmeric powder
   v) Pulses

4. Determination of adulterant in some common food items:

Suggested Readings:

1. F.W. Fifield and D. Kealy : Analytical Chemistry.
2. Daniel C Harris: Exploring chemical analysis.
SEPARATION METHODS – II

Paper IV - ANALYTICAL CHEMISTRY-4 66hrs (4h/w)
UNIT – I 12hrs

GEL, AFFINITY AND GAS CHROMATOGRAPHY
A. Gel chromatography: Principle, types of gels, separation by gel chromatography, applications.
B. Affinity chromatography: Principle, materials, selection and attachment of ligand, practical procedure, applications.
C. Gas- liquid chromatography: Apparatus and materials, preparation and application of samples, separation conditions, detectors, applications.

UNIT – II 12hrs

ELECTROPHORESIS I

Theory and classification, factors affecting mobility, macromolecular size and charge interactions with supporting electrolyte, pH and concentration discontinuities, factors affecting electrophoresis phenomena: electrolysis.

UNIT – III 12hrs

ELECTROPHORESIS II
Electro-osmosis, temperature and supporting media; instrumentation, methodology, preparation of gel-staining and de-staining, preparative zone electrophoresis, continuous electrophoresis, applications.

UNIT – IV 12hrs

DIALYSIS AND MEMBRANE FILTRATION
A. Filters- nitrocellulose, fiberglass, polycarbonate
B. General laboratory methods.

UNIT – V 12hrs

CENTRIFUGATION METHODS:
Introduction, sedimentation and relative centrifugal force, different types of rotors, density gradients, types of centrifugation techniques.
LABORATORY COURSE IV

Separation Techniques
(At the end of Semester-IV) 30 hrs (2 h / w)

1. Determination of the strength of the given HCl solution by titrating it against NaOH solution conductometrically.

2. Separation of a mixture of Ni$^{2+}$ and Cu$^{2+}$ by TLC and identify the ions.

3. Determination of residual chlorine in city water supply using colorimetry.

4. Determination of adsorption isotherm of acetic acid on activated charcoal.
   Determination of the adsorption constant (k)

5. Determination of nicotine content in cigarette tobacco.
Suggested Readings:

3. F.W. Fifield and D. Kealy: Principles and practice of analytical chemistry.

Keith Wilson and John Walker: Practical Biochemistry.

Chromatography: Basic Principles, Sample Preparations and Related Methods by Elsa Lundanes, Leon Reubsaet, Tyge Greibrokk, John Wiley and Sons, 2013
SEMESTER V

Paper – V  ANALYTICAL CHEMISTRY-5  45hrs (3h/w)
ANALYTICAL BIOCHEMISTRY AND ENVIRONMENTAL CHEMISTRY

UNIT – I
9 hrs

BASIC UNDERSTANDING OF THE STRUCTURES, PROPERTIES AND FUNCTIONS OF CARBOHYDRATES, LIPIDS, AND PROTEINS
1. Isolation and characterization of polysaccharides.
2. Classification of lipids, properties, functions and Biochemical functions of steroid hormones.
5. Enzymes- nomenclature, classification, effect of pH, temperature on enzyme activity, enzyme inhibition.
6. Lipoproteins

UNIT – II
9 hrs

BIOCHEMISTRY OF DISEASE: A DIAGNOSTIC APPROACH
Clinical chemistry: a diagnostic approach by blood/urine analysis.
1. Blood: Composition and functions of blood, blood coagulation.
2. Blood collection and preservation of samples.
3. Anemia
4. Regulation, estimation and interpretation of data for blood sugar, urea, creatinine, cholesterol and bilirubin.
5. Urine: Collection and preservation of samples.
7. Composition and estimation of constituents of normal and pathological urine.

Unit-III:
9 hrs

Microbiological Tests and Assays:
Microbiological assay of antibiotics, (std. preparations and units of activity, test organisms and inoculum, apparatus, methods: cylinder or cup plate method and two level factorial assay (ampicillin), microbial limit test (preliminary testing, medium soyabean casein digest agar medium only) and total microbial count only), test of sterility-membrane filtration method, determination of thiomersal.

Unit-IV:
9 hrs

Standardization and Quality Control of different Dosage Forms:
Brief introduction to different dosage forms with the IP requirements, analytical methods for the following: Tablets (asphiln), 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 (sulphacetamine), aerosols (salbutamol), blood products and reporting protocols.
UNIT - V

Concept and scope of environmental chemistry - nomenclature - environmental segments -
The natural cycles of the environment - the hydrological cycle - the oxygen cycle - the
nitrogen cycle.

Classification of water pollutants - Characterization - Dissolved Oxygen - BOD - COD -
Waste water treatment (General). Disposal of radioactive wastes. Pollution due to some
typical industries like Textile, Pulp and Paper, Electroplating, Dairy, Cane sugar
SEMESTER V

Paper – VI: ANALYTICAL CHEMISTRY-6 45hrs (3h/w)

INSTRUMENTAL METHODS OF ANALYSIS

UNIT –I

AN INTRODUCTION TO SPECTROSCOPIC METHOD OF ANALYSIS:
(RECAP of the introduction covered in detail in the chemistry syllabus)

UV AND VISIBLE SPECTROPHOTOMETRY:
Lambert-Beer’s law: Principles - Instrumentation, Single/double beam instrument and its applications Instrumentation – Interferences

IR SPECTROSCOPY:
Principle – Instrumentation and applications

UNIT –II

ATOMIC EMISSION SPECTROSCOPY (Flame photometry):
Principle Instrumentation – Analytical techniques for Flame photometry – Calibration plots (Working curves). Determination of Alkali and Alkaline earth metals in natural water (any two metal ions)

UNIT-III:

ATOMIC ABSORPTION SPECTROSCOPY:

Principle Radiation sources (line sources) Hollow cathode lamps and Discharge lamps. Interferences – Analytical techniques for AAS – Calibration plots. Applications – Determinations of Calcium and Magnesium in tap water. The Ilkovic

UNIT-IV:

POLAROGRAPHY AND COULOMETRY:

(a) Polarography:

(b) Coulometry: Types of coulometric methods: Potentiostatic and amperostatic; principles, instrumentation and applications, applications.

UNIT-V:

Basic Electro-analytical Chemistry:
Electrochemical cells, cell potentials, electrode potentials, calculation of cell potentials and currents in electrochemical cells, types of polarization, types of electroanalytical methods, problems.

Non-selective Electrodes:
Types of ion-selective electrodes: Glass, liquid ion exchange membrane, neutral carrier membrane, coated wire, gas sensing, air gap and biomembrane electrodes; theory of ion selective electrode and ion-selectivity coefficient; problems.

**Practical VI  Instrumental methods of analysis**  

Determination of metals in given samples by AAS technique.
Preparation of standard calibration graphs of Pb, Cd, Zn and Fe by AAS

**Suggested Readings:**

2. G.W. Castellan: Physical Chemistry.
ANALYSIS OF APPLIED INDUSTRIAL PRODUCTS

UNIT-I

ANALYSIS OF SOAPS, DETERGENTS AND PAINTS
Analysis of soaps: moisture and volatile matter, combined alkali, total fatty matter, free alkali, total fatty acid, sodium silicate, and chlorides.
Analysis of paints: Vehicle and pigments, Barium Sulphate, total lead, lead chromate, iron pigments, zinc chromate

UNIT-II

ANALYSIS OF FATS & OILS AND INDUSTRIAL SOLVENTS
Analysis of oils: saponification value, iodine value, acid value, ester value, bromine value, acetyl value.
Analysis of industrial solvents like benzene, acetone, methanol, and acetic acid.
Determination of methoxyl and N-methyl groups.

UNIT-III

ANALYSIS OF FERTILIZERS, STARCH, SUGARS AND PAPER
Analysis of fertilizers: urea, NPK fertilizer, super phosphate,
Analysis of DDT, BHC, endrin, endosulfone, malathion, parathion.,
Analysis of starch, sugars, cellulose and paper,

UNIT-IV

ANALYSIS OF GASES
Gas analysis: carbon dioxide, carbon monoxide, oxygen, hydrogen, saturated hydro carbon, unsaturated hydrocarbons, nitrogen, octane number, cetane number
Analysis of Fuel gases like: water gas, producer gas, kerosene (oil) gas.
Ultimate analysis: carbon, hydrogen, nitrogen, oxygen, phosphorus and sulfur,

UNIT-V

ANALYSIS OF COMPLEX MATERIALS:
Analysis of cement: loss on ignition, insoluble residue, total silica, sesqui oxides, lime, magnesia, ferric oxide, sulphuric anhydrid.
Analysis of glasses: Determination of silica, sulphur, barium, arsine, antimony, total R₂O₃, calcium, magnesium, total alkalies, aluminium, chloride, fluoride

Practical-VII
Preparation of soaps and detergents.
Estimation of EDTA in detergent and shampoo.
3. Analysis of Applied Industrial Products
4. Analysis of Heavy & Fine Chemicals:
5. Assay of soaps and detergent
6. Determination of Na/K/Li/Ca in given sample by flame photometry method.
7. Determination of washing strength of detergents by surface tension method.
   Determination of CMC of detergents.
   Preparation and characterization of copper sulphate.
8. Preparation and characterization of methyl orange and methyl red.
9. Estimation of Na₂CO₃.10H₂O in washing soda.
10. Determination of thiosulphate content of a commercial hypo solution.
11. Estimation of available chlorine in the sample of bleaching powder

SUGGESTED BOOKS:
1. F.J.Welcher-Standard methods of analysis,
2. A.I.Vogel-A text book of quantitative inorganic analysis-ELBS,
3. H.H.Willard and H.Deal- Advanced quantitative analysis- Van Nostrand Co,
4. F.D.Snell & F.M.Biffen-Commercial methods of analysis-D.B.Taraporewala & sons,
5. J.J.Elving and I.M.Kolthoff- Chemical analysis - A series of monographs on analytical chemistry and its applications -- Inter Scien ce- Vol I to VII,
   G.Z.Weig - Analytical methods for pesticides, plant growth regulators and
   food additives - Vols I to VII,
7. F.J.Welcher-Standard methods of analysis,
8. Quantitative analysis of drugs in pharmaceutical formulations by P.D.Sethi, CBS
   Publishers and Distributors, New Delhi
9. G.Ingram- Methods of organic elemental micro analysis- Chapman and Hall,
10. H.Wineciam and Bobbles (Henry J)- Instrumental methods of analysis of food additives,
11. H.Edward-The Chemical analysis of foods; practical treatise on the examination of food stuffs and the detection of adulterants,
SEMMESTER VI

Paper VIII

ANALYTICAL CHEMISTRY- VIII

IPR, QA & QC and Regulatory Affairs

UNIT-I:

INTELLECTUAL PROPERTY RIGHTS:
Concept and fundamentals of IPR, need and economic importance of IPR, detail
description of various IP Properties (Patents, Trademarks, Copyrights, Geographical
Indications Industrial Designs and Trade secrets). IPR with emphasis on patent
regime, factors affecting IP protection, penalties for violation or infringement, trade
related aspects of IPR, concepts behind GATT, WTO, TRIPS, TRIMS and GATS.

UNIT-II:

R & D AND TECHNOLOGY TRANSFER:
Role of R&D, functional structure of R&D, unit research strategies and
manufacturing interface, laboratory-industry interface, technology transfer
Pilot Plant Operation and Scale up:
Purpose planning, design and operation, analysis of results, assessment of flexibility of
design comprises to cope-up for safety and economic in construction and operation.

UNIT-III:

QUALITY CONTROL:
Concept of quality and quality control, nature of variability’s, design of QC laboratory for
chemical, instrumental and microbiological laboratories, schedule L1, standardization
of reagents, labeling of reagents, control samples, data generation and storage, QC
documentation, LIMS sampling techniques, sampling plans, steps to improve quality with
reference to ISO and TQM, preparation of control charts, sampling, inspection, cost
reduction & quality improvement.

UNIT-IV:

QUALITY ASSURANCE:
Concepts of Quality Assurance, Total Quality Management, Philosophy of GMP and
cGMP, preparation of audit, Conducting audit, Audit Analysis, Audit Report and
Audit follow up. Premises: Location, design, plant layout, construction, maintenance of
sterile areas, control of contamination.

UNIT-V:

INDUSTRIAL STANDARDS AND CONTROL:
Government standards like Agmark, Hallmark, ISI, MINAS, IP, BP, USP; an
introduction of ISO, OSHA, CDSCO, USFDA, ICH, FPO, MHRA, SUPAC

PROJECT WORK

Four weeks duration in any industry / chemical R&D / organization
Each candidate has to submit a project report after completion.
Suggested Books:
- Handbook of Small Scale Industry by P.M. Bhandari.
- Dr. T Ramakrishna -Ownership and Enforcement of Intellectual Property Rights, CIPRA, NSLIU-2005.
- The Trade marks Act 1999 (Bare Act with short comments) - Universal Law Publishing Co. Ltd. 2005.
- The Patents Act, 1970 (Bare Act with short comments) - as amended by Patents (Amendment) Rules 2006 w.e.f. 5-5-2006. Commercial law publishers (India) Pvt. Ltd. 2006.