

**VEER NARMAD SOUTH GUJARAT UNIVERSITY**  
**M.Sc. (ANALYTICAL CHEMISTRY)**  
**TO COME IN FORCE FROM JUNE-2011 (REVISED IN B O S Dated 18-01-2011)**  
**Practical Syllabus**

**Semester III**

**Classical methods of Analysis**

1. Analysis of ores such as Haematite, Dolomite, Lime stone, Bauxite and Pyrolusite (Minimum 3 to be done) for their major constituents.
2. Analysis of Portland cement for its calcium, magnesium and silica content.
3. Analysis of organic materials: Glycerol, Formalin, Dye intermediate, Glycine, Ethylene glycol, Amines, Nitro compounds.
4. Determination of  $Pb^{+2}$  as  $PbCrO_4$  after precipitation from homogeneous solution and  $Ba^{+2}$  as  $BaSO_4$ .
5. Analysis of bleaching material.
6. Determination of COD of water sample.
7. Determination of DO of water sample.

**Electrical methods of Analysis**

1. Potentiometric determination of  $Cl^-$  and  $I^-$  in a mixture.
2. Determination of  $Ka_1$  and  $Ka_2$  of phosphoric acid.
3. Determination of fluoride using Iron-Selective electrode.
4. Conductometric determination of vanillin in Vanilla.
5. Polarographic determination of  $Cd^{+2}$  and  $Zn^{+2}$  in a mixture.

**Optical and Separation methods of Analysis**

1. Analysis of APC tablets for its aspirin and phenacetin content using UV spectrophotometry.
2. Analysis of Barbiturates using UV spectrophotometry.
3. Simultaneous determination of  $Cr^{+3}$  +  $Co^{+2}$  in a mixture.
4. Photometric titration of  $(Cu^{+2} + Ca^{+2})$  in a mixture.
5. Determination of Nitrite.
6. Determination of Iron in brass and dolomite.
7. Biuret in the sample of urea
8. Paper chromatographic separation.
9. Determination of total salt content using IEC.

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**Practical Syllabus**

**Semester IV**

**Classical methods of Analysis**

1. Analysis of alloys:  
(A) Cu, Zn and Fe in brass.  
(B) Cu, Ni in german silver.  
(C) Manganese in steel Spectrophotometrically.
2. Analysis of Drugs:  
(A) Sulpha drugs by non-aqueous titration and argentometric titration. (B) Novalgin  
(C) Antacid tablets.  
(D) Analysis of Penicilin  
(E) Paracetamol  
(F) Iron formulation for iron content.
3. Analysis of Insecticides: Analysis of BHC.
4. Determination of Protein content of wheat flour.
5. Analysis of Detergent sample for  $\text{PO}_4$  and other constituents.
6. Analysis of fertilizers by determination of nitrogen content.
7. Analysis of fruit juice for Vitamin-C.

**Electrical methods of Analysis**

1. Amperometric titration of (i)  $\text{Pb}^{+2}$  with  $\text{K}_2\text{Cr}_2\text{O}_7$  and (ii)  $\text{Ni}^{+2}$  with D.M.G.
2. Constant current Coulometric titration of (i)  $\text{As}_2\text{O}_3$  (ii) Phenol.
3. Biamperometric titration of Iodine with  $\text{Na}_2\text{S}_2\text{O}_3$ .
4. Analysis of dye intermediate containing  $-\text{NH}_2$  by Potentiometric titration.
5. Electogravimetric determination of  $\text{Cu}^{+2}$  in brass.
6. Conductometric determination of mixture of acids (strong and weak acid)

**Optical and Separation methods of Analysis**

1.  $\text{pK}_{\text{In}}$  of indicator and determination of Isobestic point.
2. Determination of Phosphate.
3. Determination of Metal: Ligand ratio in complex.
4. Flame photometric determination of  $\text{Na}^+$  and  $\text{K}^+$ .
5. Sugar in Blood.
6. Quinine by fluorescence method.
7. Determination of Manganese in steel.
8. Silica and  $\text{Pb}^{+2}$  with dithiazone in a water effluent.
9. TLC separation.
10. Ion exchange separation of ( $\text{Fe}^{+3} + \text{Co}^{+2}$ ) and determination of  $\text{Fe}^{+3}$  colorimetric.
11. Interpretation of IR, NMR, Mass Spectra (Dry lab)

**Note:**

- Practical examination will be for **2 days in each semester.**
- **6 hours** duration on each day.

**VEER NARMAD SOUTH GUJARAT UNIVERSITY**

**M.Sc. Part-II ANALYTICAL CHEMISTRY**

TO COME IN FORCE FROM JUNE-2011

**PAPER-I**

**AC-301**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-III**

**(ELECTROANALYTICAL TECHNIQUES)**

**UNIT-I: BASIC ELECTRONICS:**

**(15 Periods)**

Introduction, Instruments for Analysis, Data Domains, Nonelectrical and electrical domains, Detectors, Transducers and Sensors, Review of electrical components, laws of electricity, Kirchhoff's laws, DC current voltage and resistance measurements, AC circuits, semiconductor and semiconductor devices, Readout devices, Operational amplifiers, Their applications to various measurement devices, current and voltage control, mathematical operations servomechanisms, Digital electronics, Logic gates (Interdomain conversions).

**UNIT-II: ELECTRICAL METHODS BASED ON CURRENT**

**(15 Periods)**

Electrolysis, Electrogravimetry, Factors affecting the quality of deposits, Applications. Principle of Coulometry, Controlled potential coulometry, Coulometric titrations (primary and secondary) applications, Acid-base titrations, Determination of arsenite, Mercaptants, Phenol.

**UNIT- III: VOLTAMETRY**

**(15 Periods)**

**Rapid Scan Voltammetry:** Principle, Rapid voltage scan at the end of the drop life, Peak current equation, Relation of peak current with the scanning rates, Summit potential equation, Comparison with DC polarography, Limitations.

**Hydrodynamic Voltammetry:** Principle and similarity with dc polarography, Types of electrodes used, Applications of the technique in determination of rate constant of the reaction.

**Anodic Stripping Voltammetry:** Concentration and stripping steps, Importance of Hanging mercury drop electrode and MTFE, Sensitivity of the technique, Adsorptive stripping, Applications, Cathodic stripping.

**Cyclic Voltammetry:** Principle, Forward and reverse scan, cyclic voltamogram, Detection limits, Applications.

**Books:** On last page.

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**M.Sc. Part-II ANALYTICAL CHEMISTRY**  
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**PAPER-I**

**AC-401**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-IV**  
**(ADVANCE ANALYTICAL TECHNIQUES)**

**UNIT-I: SURFACE SPECTROSCOPY (15 Periods)**

- (i) Introduction, Types of surface measurements, General techniques in surface spectroscopy, Sampling surfaces, Surface contamination, **Electron Spectroscopy:** Auger electron spectroscopy, Ion Spectroscopy, Surface Photon and Electron stimulated methods, Instrument and applications.
- (ii) **Atomic Fluorescence Spectroscopy:** Principle, Apparatus, Source, Cells, Wavelength selectors, Analysis with APS.

**UNIT-II: ADVANCE CHROMATOGRAPHY (15 Periods)**

- (i) **Supercritical Fluid Chromatography (SFC):** Properties of supercritical fluid, SFC, SF-extraction.
- (ii) **Electrophoresis:** Zone-Electrophoresis: Moving boundary electrophoresis, Zone electrophoresis, Continuous (Curtain) flow electrophoresis, Gel electrophoresis. Capillary Electrophoresis, Column, Electro Chromatography, Field Flow Fraction (FFF), Application.

**UNIT-III: HYPHENATED TECHNIQUES AND AUTO ANALYSERS (15 Periods)**

**Mass Spectral methods:** MS-FTIR, MS- NMR, ICP-MS, **Chromatography- Mass:** GC-MS, LC-MS, **Mass-Mass:** MS-MS, Tandem Mass Spectra, **Chromatography- Spectral:** GC-FTIR, TG- FTIR, TG-MS. Voltametric Sensors (FIA). Optical Sensors, Microprocessors. **Autoanalyser:** Need for autoanalyser, Instrument used in clinical laboratory, Flow Injection Analysis, Micro Fluid Disk, Discreet Automatic System.

**Books:** On last page.

## REFERENCE BOOKS:

- (1) Instrumental Analysis: G. D. Caristian and J. E. O'Reilly (Allyn & Bacon Inc., New York, 2<sup>nd</sup> edition.
- (2) Instrumental Methods of Chemical Analysis: G. W. Ewing (McGraw-Hill, New York), 5<sup>th</sup> edition.
- (3) Instrumental Methods of Analysis: H. R. Willard, L. L. Merrit, J. A. Dean, F. A. Settle (Van Nostrand Reinhold Co., New York), 6<sup>th</sup> edition.
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- (6) Analytical Chemistry: Principles and Techniques: Larry G. Hargis (Prentice-Hall International edition).
- (7) Introduction to Modern Liquid Chromatography: L. R. Shyder & J. J. Kirkland (John Wiley & Sons, New York).
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- (14) Environmental Chemistry: B. R. Sharma, H. Kaur (Goel Publishing House, Meerut).
- (15) Inorganic Quantitative Analysis: A. I. Vogel (Orient Longman).
- (16) "Polarography", J. D. Talati (In Gujarati), University Granth Nirman Board.
- (17) "Polarography": Kolthoff I. M. and Lingane J. J. (Vol. I & II) (Interscience Publishers, New York).
- (18) "Polarographic Techniques": L. Meites (Interscience Publishers, New York).
- (19) Principles of Instrumental Analysis (5<sup>th</sup> ed.) by Skoog, Holler and Nieman (Saunders College Publishings).
- (20) Undergraduate Instrumental Analysis (5<sup>th</sup> ed.), J. W. Robinson (Marcel Dekker Inc.).
- (21) Fundamentals of Molecular Spectroscopy, by Banwell.
- (22) Electronic Absorption Spectroscopy and related techniques, D.N. Sathyanarayan, (New Age International ND. 1996) Uni. Press, Hyderabad.
- (23) Introduction to Spectroscopy (3<sup>rd</sup> ed.) by Pavia Lampman Kriz, Cengage Learning Harcourt College Publishers.

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**PAPER-II**

**AC-302**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-III  
(SPECTROSCOPY)**

**UNIT-I : IR AND RAMAN SPECTROSCOPY (15 Periods)**

Theory of IR and Raman, selection rules, IR absorption, Raman scattering, Mutual exclusion rule, complimentary techniques, Instrumentation - FTIR and Raman, Cells and sampling techniques, Resonance Raman spectroscopy, Interpretation of IR spectra using correlation charts, Advantages of FTIR spectroscopy, Mid-IR Reflection – DRS, ATR, Data processing in Near IR, Applications in structure elucidation of inorganic and organic molecules.

**UNIT-II : NMR SPECTROSCOPY (15 Periods)**

Theory of NMR, Relaxation, population of energy levels, Larmor precession, chemical shift and factors affecting it, references and solvents, Spin-spin splitting, Coupling constant, Magnetic Anisotropy, Instrumentation, Shift Reagents, Interpretation of simple NMR spectra, Signal averaging, FT-NMR, Pulse FT-NMR spectroscopy, <sup>13</sup>C NMR spectra, NMR in medical diagnostics, Double resonance technique, Multi dimensional NMR, Problems to elucidate structure from NMR spectra.

**UNIT-III : MOLECULAR MASS SPECTROSCOPY (15 Periods)**

Instrumentation, Methods of ion production (EI, CI, FI, FD, Electro Spray, MALDI), Ion separators, Ion collection and recording, Double focusing, Time of flight analyser, Quadruple-mass spectrometer, Sample handling techniques, Resolution, Parent peak, Base peak, Metastable ions isotope effect, Molecular formula from mass spectra, Nitrogen rule, Ring rule, Fragmentation rules, Behavior of classes of compounds, Interpretation of mass spectra, Additional applications, Problems to elucidate structure from mass spectral data.

**Books:** On last page.

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**M.Sc. Part-II ANALYTICAL CHEMISTRY**

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**PAPER-II**

**AC-402**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-IV  
(SPECTROSCOPY)**

**UNIT-I: ATOMIC X-RAY SPECTROSCOPY (15 Periods)**

Emission of X-ray, continuum and line spectra, X-ray absorption, absorption spectra, Apparatus, Source (monochromatic X-ray), Sample handling, Wavelength and energy dispersive device, Detector, Chemical analysis by X-ray absorption, X-ray fluorescence: Theory, instrumentation and applications, X-ray diffraction: Theory, instrumentation and applications.

**UNIT-II: ATOMIC SPECTROSCOPY (15 Periods)**

**(a) Atomic Absorption Spectroscopy (AAS)**

Principle of AAS, Instrument, Continuous sources and line sources, Flames, Flame atomizers, Non flame atomizers (furnaces), Monochromator and Detector, Interference with AAS Quantitative Analysis with AAS, Applications, Numerical.

**(b) Flame Emission Spectroscopy (FES)**

Flame as a source of atomic vapour, Flame atomization, Flame photometer, Applications and limitations comparison with AAS

**(c) Plasma and Electrical Discharge Emission**

Emission spectroscopy with plasma sources, Instrument, AES with electrical discharge, Electrodes of AES, DC- arc, spark, Laser microprobe, Salient features of the emission spectrograph, Qualitative and Quantitative analysis applications.

**UNIT-III: ESR & APPLICATION OF UV- VISIBLE (15 Periods)**

**(a)** Theory of ESR, Population of E levels, relaxation time, Larmor precession, Instrumentation of ESR, Analytical applications of ESR spectroscopy, Hyperfine coupling mechanism, Super hyperfine splitting, g factor, Fine structure in ESR spectra hyperfine splitting constant, applications of ESR in study of metal complexes.

**(b)** Application of UV- Visible, Photometric accuracy, Simultaneous determinations of two components in a mixture,  $pK_{In}$  of indicator, Photometric titrations, Determination of metal to ligand ratio, analysis of drugs by UV-VIS spectrophotometry, Quantitative analysis, Numericals.

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TO COME IN FORCE FROM JUNE-2011  
**PAPER-III**  
**AC-303**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-III**  
**(ELECTROANALYTICAL TECHNIQUES)**

**UNIT-I: DC-POLAROGRAPHY**

**(15 Periods)**

Principle, Types of currents, Electro capillary maxima, Maxima suppressors, Interference of oxygen DME as electrode, Wave equation, Ilkovic equation (derivation), half wave potential, Applications, Determination of stability constants of complexes. Quantitative analysis, Numerically, Applications of DC polarography, limitations, **Amperometric titrations:** Principle, DME & RPE, curves, Biamperometric titration.

**UNIT-II: MODERN POLAROGRAPHIC METHODS**

**(15 Periods)**

Electrochemical Definitions and Terminology, Faradic and Non-Faradic processes, Concentration profiles at microelectrode surface during electrolysis:

**A.C. Polarography:** Principle of Sinusoidal alternating applied potential, AC peak polarogram, Peak current equation, Characteristic of AC polarographic peak, Importance of signal to noise ratio for the sensitivity, Comparison with DC polarography.

**Square-wave Polarography:** Principle of alternating rectangular wave voltage applied, Frequency of square wave applied, Problems of large condenser currents in A.C., Peak polarogram, Peak current equation, Limitations of techniques.

**Pulse Polarography:** Effect of capillary response with frequency of applied square wave potential, Principles and difference between Normal Pulse Polarography and Differential Pulse Polarography, Importance of charging and Faradaic currents.

**UNIT-III: POTENTIOMETRIC METHODS OF ANALYSIS**

**(15 Periods)**

Membrane potential, Principles of selectivity, Methods of evaluating selectivities separate and mixed solution method. Classification of ion selective electrodes, Solid state electrodes – Glass electrode effect of glass structure on selectivity function of the glass electrode. Acid error, Alkali error, Silver halide, Sulphide, Lanthanum fluoride ion selective electrodes. Liquid ion exchange electrode – Calcium selective ion electrodes. Gas electrodes, ammonia, sulphur dioxide, oxygen and CO<sub>2</sub> sensing electrode, Micro ion selective electrode, enzyme electrodes. Quantitative determination – known addition method – known subtraction method.

**Books:** On last page.

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**PAPER-III**  
**AC-403**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-IV**  
**(SEPARATION TECHNIQUES)**

**UNIT-I: SOLVENT EXTRACTION (15 Periods)**

Review of distribution law, Distribution coefficient, Distribution ratio, Equations for the solute dissociating or associating in one phase, Successive extractions, Extraction of metal ion with chelating agent with necessary equation, Multiple extractions, Craig pseudo counter current extractions, Apparatus for Craig extractions, Continuous counter current extractions, Extraction involving association of ion pairs, Elementary idea of extraction with crown ethers, Cryptans, Use of organic reagents, Numericals.

**UNIT-II: THEORY OF CHROMATOGRAPHY: PROCESS (15 Periods)**

**ANALYSERS**

**Theory of Chromatography:** Methods of elution, Ideal and non-ideal chromatography, Plate theory, Rate theory, Reasons for broadening of lands, Van Deemter equation and significance of terms involved, Optimum velocity, Resolution, Methods to improve resolution, GLC, Supports for liquid stationary phases, Selection of columns, FSOT, Selective Detectors- FPB, TID, Temperature programming in GC, Derivatisation in GC, Qualitative analysis from retention parameters, Quantitative analysis, Headspace Analysis, Thermal Desorption.

**UNIT-III: LIQUID CHROMATOGRAPHY: (15 Periods)**

**(a) Liquid Chromatography:**

Principle of HPLC, Instrument and significance of each component, Pumps, Guard column, Stationary phases (solid, liquid), Bonded phase supports, Detectors UV absorption, Fluorescence detector, RI detectors, electrochemical detectors, Normal phase and Reversed phase.

**(b) Ion-exchange Chromatography:**

Resins used, Principle of exchange, Factors affecting the exchange, Capacity of resin and its determination, Techniques, IEC with eluent suppressor columns, Applications.

**(c) Gel-permeation Chromatography:**

Principle, Types of gels, Theoretical principles, Techniques and applications.

**(d) Plane Chromatography: Paper and TLC:** Two dimensional, solvent systems, Location, HPTLC and applications.

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**PAPER-IV**

**AC-304**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-III  
(APPLIED ANALYSIS)**

**UNIT-I: ANALYSIS OF ORES, ALLOYS AND CEMENT (15 Periods)**

**(a) Ores:** General methods for the analysis of ores with special reference to the ores of iron, copper, aluminium, manganese, chromium, titanium, calcium, magnesium (for determination of trace elements, instrumental methods to be stressed).

**(b) Alloys:** Analysis of brass, German, silver, bronze, stainless steel, Monels, Ferromanganese, Alloys of Al, Mg and Ti (Emphasis should be given on instrumental methods such as AAS, molecular spectrophotometry, fluorescence, emission, spectroscopy for analysis of trace elements).

**(c) Cement:** Composition of cement and characterization, setting and hardening of cement, Analysis of cement for silica, calcium, magnesium, iron, sodium and potassium using ISI method.

**UNIT-II: ANALYSIS OF WATER, AIR, SOIL (15 Periods)**

**(a) Water:** Sources of water pollution, Sewage and industrial effluents, Analysis of water pollutants, Sampling, Preservation, Measurement of parameters such as COD, BOD, DO, TDS, suspended solids, TCC, phenols, fluoride, nitrite, sulfide, cyanides, heavy metals.

**(b) Air:** Sampling, Analysis of air borne particulates using emission spectroscopy, Determination of CO, SO<sub>2</sub>, CO<sub>2</sub>, NO<sub>x</sub>, H<sub>2</sub>S, O<sub>3</sub> in air sample. Non-dispersive IR spectrophotometry to determine CO and CO<sub>2</sub>.

**(c) Analysis of Soil:** Moisture, pH, Total nitrogen, phosphorous, silica, lime, magnesia, sulfur, manganese.

**UNIT-III: ANALYSIS OF DRUGS AND FOOD PRODUCTS: (15 Periods)**

**(a) Drugs:** Analysis of sulfa-drugs, Analgesics, Antipyretics, Antimalarial, Antiallergic (Anti-histamines), Antibiotics (Emphasis should be given on the methods given in pharmacopeia), Narcotics.

**(b) Clinical Chemistry:** Determination of glucose, electrolytes, urea, cholesterol, uric acid in blood serum.

**(c) Food Products:** Analysis of (i) Oils and fats, Iodine value, Saponification value, RM value, (ii) reducing and non-reducing sugars (iii) butter, honey, fruit, juices, non-alcoholic beverages, (iv) adulteration in oil, ghee, butter.

**Books:** On last page.

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**PAPER-IV**

**AC-404**

**Max. Marks: 70**

**Total Periods: 45**

**SEMESTER-IV**

**(APPLIED ANALYSIS)**

**UNIT-I: ANALYSIS OF SOAPS, DETERGENTS AND AGROCHEMICALS**

**(15 Periods)**

**(a) Soaps and Detergents:** Classification of detergents, Action of detergents, Determination of alcohol soluble materials, moisture, active constituents, silicates, phosphates, borates etc.

**(b) Analysis of Moisture and Biuret in urea sample,** Determination of urea nitrogen using titrimetric (urease method), Ammonical and nitrate nitrogen with Devarda's method, Total phosphorous with alkalimetric ammonium molybdate method and spectrophotometric method, K using flame photometry.

**(c) Pesticides:** Determination of total chlorine in chloride containing pesticides, Total phosphorous in phosphorous containing pesticides, Determination of traces of pesticides using GC and HPLC, Determination of Aldrin using IR spectrophotometry.

**UNIT-II: THERMAL METHODS OF ANALYSIS**

**(15 Periods)**

Thermogravimetry, Instruments for TGA & DTG, Calibration of temperature scale, Factors affecting TGA results, Applications, Evolved gas detection and analysis, Differential thermal analysis (DTA), Differential scanning calorimetry (DSC), Instrument, Reference materials, Diluents, Factors affecting DTA results, Applications, Thermometric Titration (TT), Advantages, Instrument, Direct Injection Enthalpimetry, Applications of TT and DIE.

**UNIT-III: RADIO-CHEMICAL METHODS OF ANALYSIS**

**(15 Periods)**

Interaction of radiation with matter, Units of radioactivity, Statistic of counting, Background corrections, Neutron activation analysis, Sources of neutrons, Theory of instrumental neutron activation analysis, Experimental considerations, Isotope dilution analysis (Direct and Inverse), Radioimmuno assay, Radiometric titrations, Radio release methods, Radiation safety, Numericals.

**Books:** On last page.

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