VEER NARMAD SOUTH GUJARAT UNIVERSITY
M.Sc.-II (CHEMISTRY)
TO COME IN FORCE FROM JUNE-2008

Analytical Chemistry

PAPER-I (Instrumental Methods of Analysis)

Max. Marks:70 **Total Periods: 120**

SECTION – I

UNIT-I : IR AND RAMAN SPECTROSCOPY: (20 Periods)

Review of the theory of IR and Raman spectroscopy selection rules – Raman scattering – Resonance Raman spectroscopy – Outline of the instrument for Raman spectroscopy – Mutual exclusion rates, Applications.

Instrument for IR spectroscopy – Sources, Detectors, monochromators – Cells and sampling techniques – Interpretation of IR spectra using correlation charts – Quantitative analysis – FT-IR spectroscopy; Instrument, Advantages.

Problems to elucidate structure from IR spectra.

Comparison of two techniques.

UNIT-II : NMR AND ESR SPECTROSCOPY: (20 Periods)

Theory of NMR – Relaxation – Chemical shifts – References and solvents – Spin-spin splitting – Magnetic Anisotropy – Instrumentation – Shift Reagents – Interpretation of simple NMR spectra – 13C NMR spectra – Signal averaging, FT-NMR, Pulse FT-NMR spectroscopy. Problems to elucidate structure from NMR spectra (minimum 10). ABX, AMX, ABC, A2B2 Systems – NMR in medical diagnostics.

Theory of NMR – Instrumentation of ESR – Analytical applications of ESR spectroscopy – Hyperfine coupling mechanism (only qualitative).

UNIR-III : MASS SPECTROSCOPY: (20 Periods)

Instrumentation – Methods of ion production – Ion separators – Ion collection and recording – Sample handling techniques – GC-MS coupling – Resolution – Parent peak – Base peak – Metastable ions isotope effect – Molecular formula from mass spectra – Nitrogen rule, Ring rule – Fragmentation rules – Behaviour of classes of compounds – Interpretation of mass spectra – Additional applications – Problems to elucidate structure from mass spectral data.

SECTION – II

UNIT-IV: X-RAY SPECTROSCOPY AND ATOMIC FLUORESCENCE SPECTROSCOPY AND SURFACE SPECTROSCOPY: (20 Periods)

(a) **X-ray absorption** – Apparatus – Source – Sample handling – Wavelength and energy dispersive device – Detector (any one). Chemical analysis by X-ray absorption – X-ray fluorescence – Apparatus for X-ray fluorescence – Applications of X-ray fluorescence.

(b) **Atomic Fluorescence Spectroscopy:**
Principle – Apparatus – Source – Cells – Wavelength selectors – Analysis with APS.

(c) Surface characterization by spectroscopy: Introduction – Types of surface measurements. General techniques in surface spectroscopy – Sampling surfaces – Surface contamination – Auger electron spectroscopy, Instrument and applications.

UNIT-V : ATOMIC SPECTROSCOPY: (20 Periods)

(a) Flame Emission Spectroscopy:
Flame as a source of atomic vapour – Flames Atomisation – Flame photometer – Applications and limitations comparison with AAS.

(b) 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.

(c) Atomic Absorption Spectroscopy (AAS):
Principle of AAS – Instrument – Continuous sources and line sources – Flames – Flame atomizers - Nonflame atomizers (furnaces) – Monochromator and Detector – Interference with AAS Quantitative Analysis with AAS – Applications – Numericals.

UNIT-VI : BASIC ELECTRONICS: (20 Periods)

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

Reference Books:

1. Principles of Instrumental Analysis (5th Ed.) by Skoog, Holler, Nieman (Sanuder CollegePublishing) **(Chapters 1, 2, 3).**
2. Instrumental Methods of Chemical Analysis (5th Ed.) by G. R. Ewing (McGraw Hill).

NOTES: (1) From each unit one question should be set.
(2) The marks of question and length of question should be as per the weightage of the unit.
(3) Numericals are to be taught and asked wherever necessary.

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Analytical Chemistry
PAPER-II (Instrumental Methods of Analysis)

Max. Marks: 70 **Total Periods: 120**

SECTION – I

UNIT-I : POTENTIOMETRIC METHODS OF ANALYSIS: (20 Periods)

Classification of ion selective electrodes – Glass electrode – Crystalline membrane electrode – Liquid ion-exchanger electrode – Neutral carrier membrane electrode – Gas permeable membrane electrode – Biocatalytic membrane electrodes – Selectivity coefficients – Quantitative Analysis using ISE – Standard addition, Standard substraction methods – Dillution method – Double known addition methods – Ionimeter – Numericals.

UNIT-II : MODERN POLAROGRAPHIC METHODS: (20 Periods)

Electrochemical Definitions and Terminology – Faradaic and Non-Faradaic processes – Concentration profiles at microelectrode surface during electrolysis.

(A) 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.

(B) 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.

(C) 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. Instrumentation.

(D) Hydrodynamic Voltammetry:

Principle and similarity with dc polarography – Types of electrodes used – Applications of the technique in determination of rate constant of the reaction.

(E) Anodic Stripping Voltammetry:

Concentration and stripping steps – Importance of Hanging mercury drop electrode and MTFE – Sensitivity of the technique – Applications – Cathodic stripping.

(F) Cyclic Voltammetry:

Principle – Forward and reverse scan, cyclic voltamogram – Detection limits – Applications.

(G) 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.

UNIT-III : THERMAL METHODS OF ANALYSIS: (20 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.

SECTION – II

UNIT-IV : (20 Periods)

(a) Radio-Chemical Methods of Analysis:

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.

(b) Photo-acoustic Spectroscopy:

Principle and applications.

UNIT-V : THEORY OF CHROMATOGRAPHY, GC AND PROCESS ANALYSERS: (20 Periods)

(A) 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.

(B) Gas Chromatography:

Mobile phase and criteria for its selection – Sample introduction techniques – Stationary phases used in GSC and GLC – Supports for liquid stationary phases – Selection of columns – packed, WCOT, SCOT, FSOT – Detectors FID, TCD, FPB, ECD, TID – Their merits and demerits – Temperature programming in GC – Derivatisation in GC – Qualitative analysis from retention parameters – Quantitative analysis.

(C) Process analysers and used for moisture, oxygen determination – Process GC – non-dispersive IR spectrophotometer to determine CD and CO_2.

UNIT-VI: LIQUID CHROMATOGRAPHY AND AUTOCANALYSERS: (20 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:

(i) Paper chromatography (Ascending, Descending, Radial, Two dimensional) solvent systems – Location.

(ii) Thin-layer chromatography: Preparation of plates – Adsorbants and solvent systems used – Techniques of zone detection – Elementary idea of HPTLC.

(e) Zone-Electrophoresis:

Moving boundary electrophoresis – Zone electrophoresis – Continuous flow electro-phoresis – Gel electrophoresis.

(f) Autocanalysers:

Need for autoanalyser – Instrument used in clinical laboratory.

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Analytical Chemistry
PAPER-III (Applied Analysis)

Max. Marks:70 **Total Periods: 120**

SECTION – I

UNIT-I : ANALYSIS OF ORES, ALLOYS AND CEMENT (20 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 POLLUTANTS AND AIR POLLUTANTS

(20 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_2, CO_2, NO_x, H_2S, O_3 in air sample.

Non-dispersive IR spectrophotometry to determine CO and CO_2.

(c) Analysis of Soil:

Moisture, pH, Total nitrogen, phosphazous, silica, lime, magnesia, sulfur, manganese.

UNIT-III : ANALYSIS OF DRUGS AND FOOD PRODUCTS: (20 Periods)

(a) Drugs:

Analysis of sulfa-drugs, Analgesics, Antipyretics, Antimalarial, Antiallergic (Anti-histamines), Antibiotics (Emphasis should be given on the methods given in pharmacoepia), 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 bevearages, (iv) adulteration in oil, ghee, butter.

SECTION – II

UNIT-IV: ANALYSIS OF SOAPS, DETERGENTS AND AGROCHEMICALS:

(20 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 (ureas method) – Ammonical and nitrate nitrogen with Devarda's method –

Total phosphorous with alkalimetric ammonium molybdate method and spectrophoto-Metric 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-V : ANALYSIS OF FUELS AND POLYMERS: (20 Periods)

(a) Fuels:

Classification of coal – Proximate and ultimate – Analysis of coal and their significance – Bomb colorimeter to determine calorific value of solid and liquid-fuel – Boy's method to determine calorific value of gaseous fuel – Analysis of fuel gases using Orsat's apparatus. Octane number – Liquid fuels – Flash point – Aniline point.

(b) Polymers:

Number average and weight average molecular weights – Determination of molecular weight using osmometry, viscosity and light scattering.

Characterisation of polymers – The glassy state and determination of glass transition temperature – Crystallisation and melting – Determination of Tm – Using of IR and NMR for chemical characterization of polymer.

REFERENCES:

(1) Engineering Chemistry by Jain & Jain, Dhanpatrai pub. Co., Delhi.
(2) Text-book of Polymer Science, F. W. Billmeyer Jr., Pub. John Wiley & Co.

UNIT-VI : CALCULATIONS IN ANALYTICAL CHEMISTRY: (20 Periods)

Calculations based on titrimetry, gravimetry, spectrophotometric, potentiometric, polarographic and coulometric methods. Problems for the analysis of industrial materials, like ores, alloys, fertilizers, drugs, insecticides etc. Calculations involved in direct and back titrations and indirect analysis, Preparation of buffer solutions of definite pH.

REFERENCE BOOKS FOR ALL THREE PAPERS OF ANALYTICAL CHEMISTRY:

(1) Instrumental Analysis : G. D. Caristian and J. E. O'Reilly (Allyn & Bacon Inc., New York, 2nd edition.

(2) Instrumental Methods of Chemical Analysis : G. W. Ewing (McGraw-Hill, New York), 5th edition.

(3) Instrumental Methods of Analysis : H. R. Willard, L. L. Merrit, J. A. Dean, F. A. Settle (Van Nostrand Reinhold Co., New York), 6th edition.

(4) Modern Methods of Chemical Analysis : Pecsok, Shield & Cairns (John Wiley), 2nd edition.

(5) Introduction to Instrumental Analysis (1987), R. D. Braun (McGraw-Hill Book Company), New Delhi.

(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).

(8) Treatise on Analytical Chemistry : I. M. Kohthoff & P. J. Elving (John Wiley & Sons, New York).

(9) Handbook of Analytical Chemistry : L. Meites (McGraw-Hill, New York).

(10) Photometric and Fluorometric Methods of Analysis : F. D. Snell (John Wiley & Sons Inc., New York).

(11) Standard Methods of Chemical Analysis : Vol. I & II (6th edition), D. Van Nostrand Co. Inc. (London).

(12) Official Methods of Analysis : Published by Association of Official Analytical Chemists, Washington.

(13) Instrumental Methods of Chemical Analysis : B. R. Sharma (Goel Publishing House, Meerut).

(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 (5th ed.) by Skoog, Holler and Nieman (Saunders College Publishings).

(20) Undergraduate Instrumental Analysis (5th ed.), J. W. Robinson (Marcel Dekker Inc.).

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Analytical Chemistry
Practicals

[Practical examination will be of 3 days duration, 7 hours on each day. The following six exercises will be asked:

(1) Classical methods of Analysis-I.

(2) Classical methods of Analysis-II.

(3) Electrical methods of Analysis-I.

(4) Electrical methods of Analysis-II.

(5) Optical methods of Analysis.

(6) Separation methods and miscellaneous.]

Syllabus

(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 alloys:

(A) Cu, Zn and Fe in brass.

(B) Cu, Ni in german silver.

(C) Manganese in steel Spectrophotometrically.

(3) Analysis of Portland cement for its calcium, magnesium and silica content.

(4) Analysis of organic materials: Glycerol, Formalin, Dye intermediate, Glycine, Ethylene glycol, Amines, Nitro compounds.

(5) 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.

(6) Analysis of Insecticides: Analysis of BHC.

(7) Determination of Protein content of wheat flour.

(8) Determination of Pb^{+2} as $PbCrO_4$ after precipitation from homogeneous solution and Ba^{+2} as $BaSO_4$.

(9) Analysis of Detergent sample for PO_4 and other constituents.

(10) Analysis of bleaching material.

(11) Analysis of fertilizers by determination of nitrogen content.

(12) Determination of COD of water sample.

(13) Determination of DO of water sample.

(14) Analysis of fruit juice for Vitamin-C.

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) Conductometric determination of vanillin in Vanilla.

(4) Elecrtogravimetric determination of Cu^{+2} in brass.

(5) Polarographic determination of Cd^{+2} and Zn^{+2} in a mixture.

(6) Amperometric titration of (i) Pb^{+2} with $K_2Cr_2O_7$ and (ii) Ni^{+2} with D.M.G.

(7) Constant current Coulometric titration of (i) As_2O_3 (ii) Phenol.

(8) Biamperometric titration of Iodine with $Na_2S_2O_3$.

(9) Determination of fluoride using Iron-Selective electrode.

(10) Analysis of dye intermediate containing $-NH_2$ by Potentiometric titration.

Spectrophotometric methods:

(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) pK_{In} of indicator and determination of Isobestic point.

(6) Determination of Nitrite.

(7) Determination of Phosphate.

(8) Determination of Metal : Ligand ratio in complex.

(9) Determination of Manganese in steel.

(10) Determination of Iron in brass and dolomite.

(11) Quinne by fluorescence method.

(12) Biuret in the sample of urea,

(13) Silica in water.

(14) Pb^{+2} with dithiazone in a water effluent.

(15) Flame photometric determination of Na^+ and K^+.

(16) Interpretation of IR, NMR, Mass Spectra (Dry lab)

(17) Sugar in Blood.

Separation methods:

(1) Paper chromatographic separation.

(2) TLC separation.

(3) Determination of total salt content using IEC.

(4) Ion exchange separation of (Fe^{+3} + Co^{+2}) and determination of Fe^{+3} colorimetric.