

Syllabus

PG Diploma in Analytical Chemistry

Max Marks: 100 (ESE: 70 IA : 30)

Passing marks: 40

Question Paper pattern for End Session Exam (ESE)

Max Marks: 70

Part-A will contain 15 short questions of 1 marks each. Attempt ANY ten questions.

Part-B will contain 3 questions (1 from each unit with internal choice) of 20 marks each.

P011

MCH-011 Basic Analytical Chemistry

Unit-I: Extraction Methods:

Basic principles, classification of extraction systems, factors affecting extraction process, mechanism of extraction, extraction of liquids, extraction by chelation, extraction by solvation, extraction equilibria for chelates, extraction equilibria for solvation, separation of metals by extraction, extraction by ion-pair formation, solid- phase extraction (SPE), supercritical fluid extraction (SFE), supramolecular extraction, centrifugation and ultra-centrifugation, membrane separation, extraction by sonication

Unit-II: Isolation and Purification Techniques:

Filtration (simple, micro, etc.), recrystallization (in aqueous & non-aqueous solutions, at low temperature, in inert atmosphere, semi-micro & micro), use of decolourising carbon, difficulties in recrystallization, drying of liquids, freezing, sublimation, distillation (simple, steam, fractional, vacuum, high vacuum or molecular, etc.), nucleation and crystal growth, crystal hydrates and solvates, chemical methods for separation, determination of physical constants (mp, mixed mp, bp, m. wt., density, optical rotator power, RI).

Unit-III: High Frequency Titrations:

Principle, Instrumentation- Cells, oscillator circuit and high frequency titrimeters, theory, correlation of high frequency titration curves with low frequency titration curves. Applications- acid base, complexometric, measurement of dielectric constant and analysis of mixture of organic compounds. Advantages and disadvantages of high frequency methods.

P012

MCH012 Electroanalytical and Other Methods

Unit-1: Polarography:

Principles, classification of polarographic techniques, types of polarographic currents, instrumentation, factors affecting polarographic wave, pulse polarography, and differential pulse polarograph. Potentiometry: Metal electrodes for measuring the metal's cation, metal-metal salt electrodes, redox electrodes, calomel electrode, measurement of potential, determination of concentrations, residual liquid-junction potential, accuracy on direct potentiometric, glass pH electrode, ion-selective electrodes.

Unit-II: Voltammetry:

Voltammetric principles, hydrodynamic voltammetry, stripping voltammetry, cyclic voltammetry, criteria of reversibility of electrochemical reactions, quasi-reversible and irreversible processes, qualitative and quantitative analysis ,

Unit-III Amperometry:

Principles and amperometric titration techniques: Dropping mercury electrode, rotating platinum microelectrode and dead stop, biamperometric titrations.

P013

MCH 013 Advanced Analytical Chemistry

Unit-I: Thermo-analytical Methods:

Introduction and classification of thermoanalytical methods; thermogravimetric analysis (TGA): definition, types of TGA, instrumentation, TGA curve, factors affecting TGA curves, calculation of percent decomposition and composition of compounds; limitation and advantages of TGA; application of TGA to the thermal behavior including crystalline copper sulphate, calcium oxalate monohydrate, zinc hexafluorosilicate; differential thermal analysis (DTA): definition, theoretical basis of DTA, instrumentation, factors affecting the DTA curve, application of DTA, advantages and disadvantages of DTA; differential scanning calorimetry (DSC): Definition, comparison of DTA and DSC techniques, instrumentation, factors affecting DSC curves.

Unit-II: Radio-analytical Methods:

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, radio-chromatography and radioimmunoassay. Nephelometry and Turbidimetry: Introduction, theory, comparison of spectrophotometry, turbidimetry and nephelometry, instrumentation and applications

Unit-III: Polarimetry:

Polarisation of light, optical activity, theories of optical activity, factors affecting angle of rotation, specific rotation, optical rotator dispersion and circular dichroism, Cotton effect, ORD and CD curves, instrumentation, measurement of rotatory power, applications of polarimetry, optical activity and chemical constitution, representation of optical isomerism, deciding between

two structures for a molecule, distinguish between a pair of enantiomorphs, saccharimetry, difference between saccharimetry and polarimetry, saccharimeters, kinetic polarimetry, spectropolarimetry. Refractometry: Principle, parameters influencing refraction, significance of critical angle during measurements, refractometers, qualitative and quantitative analysis and analytical applications

P014

MCH 014 Analysis of Commercial Materials

Unit-I: Analysis of Petrochemicals:

Constituents, petroleum fractionation, 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 mercapton sulphur, determination of lead in petroleum. Analysis of Fuels: Proximate and ultimate analysis of fuel, calorific value by Bomb calorimetry, analysis of fuel gases (coal gas, producer gas, water gas).

Unit-II: Analysis of Agrochemicals:

Analysis of Fertilizers: Analysis nitrogen: urea nitrogen, total Kjeldahl nitrogen method, ammonia nitrogen; analysis of phosphorous: total phosphorous, available and non-available, alkalimetric ammonium molybdophosphate method; analysis of potassium: potassium by sodium tetraphenyl borate method. Analysis of herbicides: atrazine, alachlor; Analysis of Fungicides: nimbin, carbendazim; Analysis of bactericides: chloramine, triclosan, chlorhexidine; Analysis of insecticides: DDT, BHC, aldrin, endosulfan, malathion, monochrotophos; Analysis of nematicides: aldicarb; Analysis of rodenticides: warfarin, bromadiolone.

Unit-III: Analysis of Polymers:

Chemical Methods of Analysis: Introduction, preparation of the sample, determination of purity, physical tests, preliminary examination, burning characteristics, transition points, molecular weight, density, refractive index, pyrolytic behaviour, qualitative and quantitative elementary analysis, solubility and acid numbers, acetyl number, iodine number end group analysis, colour tests. Analysis of Plastics: Basics of plastic analysis, fundamental conditions for plastic analysis, water test, copper wire test, acetone test, heat test, isopropyl alcohol test, oil test.

PR01

Chemical Kinetics:

- Determination of order of reaction with respect to Ag(I) in oxidation of Mn(II) by $S_2O_8^{2-}$ and rate constant for un-catalyzed reaction.
- Determination of the primary salt effect on the kinetics of ionic reaction and testing of the Bronsted relationship (iodide ion is oxidised by persulphate ion).

- Determination of energy and enthalpy of activation in the reaction of KMnO_4 and benzyl alcohol in acid medium.
- Determination of energy of activation and entropy of activation from a single kinetic run Kinetics of decomposition of benzene diazonium chloride.
- Kinetics of decomposition of acidified hydrogen peroxide with potassium iodide and determination of activation energy. Flowing clock reactions. Oscillatory reactions.

Potentiometry / pH metry

- Determination of EMF of Daniel cell.
- Determination of standard electrode potential (E_0) value of the ferrous-ferric system by titrating ferrous ammonium sulphate against potassium dichromate potentiometrically.
- Determination of pKa of dibasic acid (oxalic acid, succinic acid, etc).
- Determination of the formation constant of Ag-ammonia complex and stoichiometry of the complex potentiometrically.
- Determination of hydrolysis constant and degree of hydrolysis of aniline hydrochloride pH metrically

PR02

Electroanalytical Methods of Analysis:

- Oxidation-Reduction Titrations
- Standardization with sodium oxalate of KMnO_4 and determination of Ca^{2+} ion.
- Standardization of ceric sulphate with Mohr's salt and determination of Cu^{2+} , NO_3^{-1} and $\text{C}_2\text{O}_4^{-2}$ ions.
- Standardization of $\text{K}_2\text{Cr}_2\text{O}_7$ with Fe^{2+} and determination of Fe^{3+} (Ferric alum)
- Standardization of hypo solution with potassium iodate / $\text{K}_2\text{Cr}_2\text{O}_7$ and determination of available Cl_2 in bleaching powder, Sb^{3+} and Cu^{2+} .
- Determination of hydrazine with KIO_3 titration. (ii) Precipitation Titrations AgNO_3 standardization by Mohr's method by using adsorption indicator. Volhard's method for Cl-determination.
- Determination of ammonium / potassium thiocyanate.
- Estimation of magnesium or cadmium as oxinate by titration with standard bromate solution. Estimation of KBr in the given solution by titrating against std. AgNO_3 solution using eosine as indicator. (iii) Complexometric Titrations:
- Determination of Cu^{2+} and Ni^{2+} by using masking reagent by EDTA titration.
- Determination of Ni^{2+} (back titration). Determination of Ca^{2+} (by substitution method). Estimation of the purity of oxalic acid employing standard Ce(IV) solution.

Analysis of Agrochemicals:

- Analysis of soil sample, soil micronutrients for Ca, Fe and P content
- Analysis of pigments with respect to Zn and Cr.
- Analysis of pesticide residue and toxicological effects.

- Analysis of malathion by colorometry.
- Determination of organic carbon in soil by Walk Ley and Black method.
- Determination of available chlorine in bleaching powder by Bunsen method.
- Determination of total chlorine in pesticide formulation.
- Determination of copper in fungicide.
- Estimation of nitrogen from given fertilizer by Kjeldahl method.
- Estimation of phosphorus from given fertilizer by volumetry / colourimetry.
- Estimation of potassium from given fertilizer by gravimetry / Flame photometry.
- Determination of K_2O content in given sample of potash fertilizer.
- Determination of P_2O_5 content in give n sample of phosphatic fertilizers.

Both the practical papers are based on the practical application of the theory papers.

Reference Books:

- D. A. Skoog and D. M. West, Fundamentals of Analytical Chemistry, Holt Rinehart and Winston Publications, IV Edn, 1982.
- D.A. Skoog, D.M. West, F.J. Holler and S.R. Crouch, Fundamentals of Analytical Chemistry, Thomson Asia Pte Ltd., Singapore, Viiiith Edn., 2004.
- D.A. Skoog, Principles of Instrumental Analysis, Saunders College Pub.Co, III Edn., 1985.
- J.G. Dick, Analytical Chemistry, McGraw Hill Publishers, 1974.
- Willard, Merit, Dean and Settle, Instrumental Methods of Analysis, CBS Publishers and Distributors, IV Edn.,1989
- G. D. Christian and J.E.O Reilly, Instrumental Analysis, Allyn and Bacon Inc, II Edn., 1986.
- G.W. Ewing, Instrumental Methods of Chemical Analysis, McGraw Hill Pub, 1975.
- Dr. B.K.Sharma, Instrumental method of Chemical analysis, Goel Publishing House, Meerut 2000
- Gurdeep R Chatwal, Sham K Anand, Instrumental Methods of Chemical Analysis, Himalaya Pub House, New Delhi 2011.