

Parvatibai Chowgule College of Arts and Science

AUTONOMOUS MARGAO - GOA

POST GRADUATE DEPARTMENT OF CHEMISTRY M. Sc. ANALYTICAL CHEMISTRY

SEMESTER III SEMESTER IV

EFFECTIVE FROM ACADEMIC YEAR

2022 - 2023

M. Sc. ANALYTICAL CHEMISTRY COURSE STRUCTURE

III AND IV	CORE COURSES		
	PGM-CHE-AC-C501: Fundamentals of Titrimetric	3	36
	Analysis		
	PGM-CHE-AC-C502: Separation Techniques	3	36
	PGM-CHE-AC-C503: Spectral Methods of Analysis	3	36
	PGM-CHE-AC-C504: Experiments in Analytical	3	72
	Chemistry		
	ELECTIVE COURSES		
	PGM-CHE-AO-E501: Advanced Mass Spectrometry	2	24
	PGM-CHE-AO-E502: Advanced NMR Spectroscopy	2	24
	PGM-CHE-AO-E503: Applied Analytical Chemistry	2	24
	PGM-CHE-AO-E504: Bio analytical Chemistry	2	24
	PGM-CHE-AO-E505: Calibrations and Validation	2	24
	PGM-CHE-AO-E506: Chemometrics	2	24
	PGM-CHE-AO-E507: Techniques in Chemical Analysis	2	24
	PGM-CHE-AO-E508: Thermal Methods of Analysis	2	24
	PGM-CHE-AO-E509: Quality Assurance and Quality	2	24
	Control in Analytical Chemistry		
	PGM-CHE-AO-D510: Dissertation	8	192
	PGM-CHE-AO-M511: Modules in Experimental Chemistry	4	96
	PGM-CHE-AO-I512: Internship Module	4	96

M. Sc. PART - II ANALYTICAL CHEMISTRY SEMESTER III AND IV

CORE COURSES

Course Code: PGM-CHE-AC-C501 **Course Title: Fundamentals of Titrimetric Analysis** Credits: 3 **Duration: 36 Hours Maximum Marks: 75**

Course Objectives:

- 1. To enable students to understand the concept of titrimetry
- 2. To enable students to understand fundamental concepts in acid-base, precipitation, complex formation, redox system

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Determine equivalence point of various titrations theoretically
- CO2: Make basic quantitative calculations regarding a number of chemical concepts
- CO3: Write and understand chemical reactions and stoichiometry

CO4: Master the concepts like chemical equilibria, complexation, solubility, acidity and basicity

UNIT I: Acid-Base Titrations

Acid-Base titrations- introduction, theory of acid-base indicators; range of indicators; selection of proper indicator; indicator errors; colour change; neutralization curves for strong acid-strong base, weak acid-strong base and weak base-strong acid weak acid-weak base titrations; poly functional acids and bases; titration curves for mono and poly functional acidsand bases; titration curves for amphiprotic species; determining the equivalence point; feasibility of acid- base titrations; magnitude of the equilibrium constant; effect of concentration; typical applications of acid-base titrations.

UNIT II: Precipitation and Redox Titrations

Precipitation titrations- introduction; feasibility; titration curves- effect of reaction completeness, effect of titrant and analyte concentration, indicators for precipitation titrations; the Volhard, the Mohr and the Fajans methods; determining the equivalence point of mixture for anions; typical applications of standard silver nitrate solution.

Redox titration- introduction, electrode potentials in equilibrium systems; equilibrium constants and its calculation; detection of end point and redox indicators, choice of indicator, structural aspect of redox indicators, specific and nonspecific indicators; equivalence point determination from redox titration curves, derivatives of titration curves; formal redoxpotentials calculation; factors affecting the shape of titration curves- concentration; completeness of reaction; feasibility of redox titrations; sample preparation- pre-reduction and pre-oxidation; applications.

UNIT III: Complexometric Titrations

Introduction; reactions of complex formation; stability of complexes; stepwise formationconstants; inorganic complexing agents- titrations involving unidentate ligands; titration of chloride with Hg²⁺ and cyanide with Ag⁺; organic complexing agents- amino carboxylic acid titration; EDTA- acidic properties of EDTA; EDTA complexes with metal ions; equilibrium

calculations involving EDTA in solution; conditional formation constants; EDTA titration curves; determining the equivalence point; effect of other complexing agents on EDTA; factors affecting the titration curves; completeness of reaction; theory of common indicators; types of titration using

12 Hours

12 Hours

EDTA, selectivity, masking and demasking agents; applications of EDTA titrations- hardness of water; magnesium and aluminium in antacids; magnesium and zinc in a mixture; analysis of ores and foods.

TEXT BOOKS:

Fundamentals of Analytical Chemistry, D. A. Skoog, D. M. West, F. J. Holler, S. R.Crouch; 8th Edition

REFERENCE BOOKS:

- 1. Principles and Practice of Analytical Chemistry, F. W. Fifield, D. Kealy; Backwell Science Ltd., London
- 2. Vogel's Text Book of Quantitative Chemical Analysis; 6th Edition
- 3. Analytical Chemistry, G. D. Christian; 5th Edition, John Wiley, NY
- 4. Instrumental Methods of Chemical Analysis, H. Kaur; Pragati Prakashan
- 5. Quality in the Analytical Chemistry Laboratory, E. Prichard; John Wiley and Sons, NY
- 6. Quantitative Analysis, R. A. Day, A. L. Underwood; Prentice-Hall

WEB REFERENCES:

- 1. https://chem.libretexts.org/Bookshelves/Ancillary_Materials/Demos%2C_Techniques %2C_and_Experiments/General_Lab_Techniques/Titration/Acid-Base_Titrations
- 2. https://opentextbc.ca/chemistry/chapter/14-7-acid-base-titrations/
- 3. https://chem.libretexts.org/Courses/Northeastern_University/09%3A_Titrimetric_Meth ods/9.5%3A_Precipitation_Titrations
- 4. https://chem.libretexts.org/Bookshelves/General_Chemistry/Map%3A_Chemistry_-_The_Central_Science_(Brown_et_al.)/17%3A_Additional_Aspects_of_Aqueous_Equi libria/17.3%3A_Acid-Base_Titrations
- 5.https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Supplemental_Modules_(Analytical_Chemistry)/Quantifying_Nature/Volumetric_Chemical_Analysis_(Shiundu)/1 4.4%3A_Complex_ion_Equilibria_and_Complexometric_Titrations

Course Code: PGM-CHE-AC-C502 Course Title: Separation Techniques Credits: 3 Duration: 36 Hours Maximum Marks: 75

Course Objectives:

- 1. To give students a theoretical and practical introduction to the techniques of separation
- 2. To address modern challenges across the chemical, biological, and physical sciences as it is often necessary to isolate and examine chemical and biological species as pure substances

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Describe the methods of separation and their applications
- CO2: Acquire technical knowledge, practical experience with respect to chromatography and capillary electrophoresis
- CO3: Understand various chromatographic techniques employed
- CO4: Learn applications of various processes for separation and purification of compounds

UNIT I: Basic Separation Techniques and Electrophoresis methods

12 Hours

General aspects of separation techniques- role of separation techniques in analysis; classification; choice of separation method; distillation- theory, principle, apparatus; operation variables and their effect; relative volatility, reflux ratio, theoretical plates and HETP; distillation columns-types and choice; applications; molecular distillation- theory; setup and applications; sublimation-

theory, principle, apparatus, applications; centrifugation and ultracentrifugation- theory, principle and applications; Electrophoresis- introduction, types of electrophoresis techniques; instrumentation and theory of capillary electrophoresis, types of capillary electrophoretic methods- capillary zone electrophoresis, micellar electrokinetic capillary chromatography; Capillary Gel Electrophoresis; Capillary electrochromatography; applications.

UNIT II: Advanced Chromatographic Techniques

12 Hours

Introduction to theory and principle of chromatographic technique; terms and parametersused in chromatography, band broadening and column efficiency; variables that affect column efficiency; Van Deemter equation and its modification; Gas Chromatography- introduction, principle, theory, instrumentation; columns in GC; detectors- ionization, flame ionization, thermal conductivity, electron capture; evaluation of gas chromatogram; identification of chromatogram; applications; comparison of GSC and GLC; applications; High Performance Liquid Chromatography- introduction; principle; instrumentation; pumps, column and column packing; column efficiency and selectivity; characteristics of liquid chromatography; types of detectors-UV, RI, and fluorescence detectors; advantages, comparison of HPLC and GLC; applications; Numerical on chromatographic parameters.

UNIT III: Miscellaneous Separation Techniques and Hyphenated Techniques 12 Hours

Gel chromatography- introduction, theory; principle of gel permeation chromatographyinstrumentation and applications; theory and mechanism of ion exclusion; applications of ion exclusion technique; inorganic molecular sieves; principle; types of sieves; applications. Supercritical Fluid Chromatography-introduction; theory, principle; properties of supercritical-fluids; instrumentation and operating variables; comparison of SFC and other column methods, applications; Field-flow fractionation- theory, mechanism, types and applications; Hyphenated Techniques- introduction; principle, instrumentation, applications of GC-FTIR; GC-MS; LC-MS, TG-MS.

TEXT BOOK:

Fundamentals of Analytical Chemistry, D. A. Skoog, D. M. West, F. J. Holler; 9th Edition

REFERENCE BOOKS:

- 1. Chemical Instrumentation: A Systematic Approach, H. A. Strobel
- 2. Analytical Chemistry, G. D. Christian; 5th Edition, John Wiley and Sons, NY
- 3. Instrumental Methods of Chemical Analysis, H. Kaur; Pragati Prakashan
- 4. Vogel's Text Book of Quantitative Chemical Analysis; 6th Edition
- 5. Instrumental Methods of Analysis, H. H. Willard, L. L. Merritt, J. A. Dean
- 6. Instrumental Methods of Chemical Analysis, B. K. Sharma; Goel Publishing House

WEB REFERENCES:

- 1. https://www.nottingham.ac.uk/-sczsteve/Ohlendieck%20and%20Harding%202018.pdf
- 2. http://www.chem1.com/acad/webtext/solut/solut-5.html
- 3. https://www.ijarnd.com/manuscripts/v2i4/V2I4-1168.pdf

4.https://www.pharmatutor.org/pharma-analysis/explain-electrophoresis-its-principle-and-factors-governing-it

5.https://www.iitk.ac.in/dordold/index.php?option=com_content&view=category&layout=bl og&id=220&Itemid=239

Course Code: PGM-CHE-AC-C503 Course Title: Spectral Methods of Analysis Credits: 3 Duration: 36 Hours Maximum Marks: 75

Course Objectives:

- 1. To provide students the basics for the materials characterization by using XRD
- 2. To enable students to understand the concept of emission measurement for quantification of related compounds
- 3. To provide students the basic knowledge about the spectroscopy for identification of inorganic compounds

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Understand the basics of emission and diffraction concepts
- CO2: Understand the different phenomenon of emission
- CO3: Understand the use of EDAX
- CO4: Apply the knowledge of Mossbauer and electron spin spectroscopy in identification of compounds

UNIT I: X-ray Absorption, Neutron Diffraction Spectroscopy

12 Hours

A. X-ray absorption- introduction, theory, origin and interaction of X-ray with matter; X-ray spectrometer; Bragg's law; interpretation of X-ray diffraction pattern; calculation of lattice parameters; neutron diffraction- introduction, theory, instrumentation and applications; X-ray fluorescence- introduction, applications; X-ray photoelectron spectroscopy.

B. Theory and interpretation of micrographs by SEM, EDAX, TEM, AFM.

UNIT II: Molecular Fluorescence, Phosphorescence and Chemiluminescence 12 Hours Fluorescence and phosphorescence- introduction, definition; principles of fluorescence, chemical structure and fluorescence; theory of molecular fluorescence; instrumentation- single and double beam filter fluorimeters; relationship between intensity of fluorescence and concentration; factors influencing fluorescence and phosphorescence; basic differences in fluorescence and phosphorescence measurement; advantages, limitations and precautions; spectrofluorometer; phosphorimeter; selection of excitation wavelength for analysis; reporting fluorescence spectra; applications of fluorimetric analysis- inorganic, pharmaceutical, agricultural, biochemical and biomedical materials: Chemiluminescence- introduction. principle. types; meaning of luminescence. chemiluminescence: chemiluminescence instrumentation: titrations. chemiluminescence measurement; chemiluminescence; phase quantitative gas chemiluminescence analysis; electro- chemiluminescence.

UNIT III: Mossbauer Spectroscopy and Raman Spectroscopy

Mossbauer Spectroscopy- introduction; principle; theory; instrumentation; line width; isomer shift; quadrupole interaction; magnetic interaction; information on spin and oxidation states; structure and bonding; spin transition from spectra of different Mossbauer active nuclei in various environments; Mossbauer effect; application of Mossbauer effect to investigate the compounds of iron and tin; Raman spectroscopy- introduction, light scattering by molecules, Raman effect- in solids, liquids, gases; mechanism; molecular structure; nature of Raman spectra; Raman activity of molecular vibrations; dynamic light scattering; determination of colloidal particle size.

TEXT BOOK:

Fundamentals of Molecular Spectroscopy, C. N. Banwell, E. M. McCash; 4th Edition, Tata McGraw-Hill, New Delhi

REFERENCE BOOKS:

1. Elements of X- ray Diffraction, B. D. Cullity; Addison Wisley

- 2. Diffraction Method, Wormald; Oxford University Press
- 3. Neutron Scattering in Chemistry, E. Butleworth Baun, G. London
- 4. Mossbauer Spectroscopy, N. N. Greenwood, T. C. Gibbs; Chapmann Hall
- 5. Chemical Application of Mossbauer Spectroscopy, V. I. Goldanski and R. H. Harber; Academic Press
- 6. Spectroscopy in Inorganic Compounds, CNR Rao, G. R. Ferraro; Academic Press
- 7. Basic Principles of Spectroscopy, R. Cheney; Mc Graw-Hill
- 8. Principles of Instrumental Analysis, D. A. Skoog, F. J. Holler; 5th Edition
- 9. Instrumental Methods of Chemical Analysis, B. K. Sharma; Goel Publishing House

WEB REFERENCES:

- 1. https://serc.carleton.edu/research_education/geochemsheets/techniques/XRD.html
- 2. https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Map%3A_Principles_of_I nstrumental_Analysis_(Skoog_et_al.)/15%3A_Molecular_Luminescence_Spectrometry
- 3. https://www.sciencedirect.com/topics/earth-and-planetary-sciences/mossbauer-spectroscopy
- 4. https://serc.carleton.edu/research_education/geochemsheets/techniques/mossbauer.html
- https://www.google.com/url?sa=t&source=web&rct=j&url=https://www.nanoscience.co m/techniques/atomic-force-microscopy/&ved=2ahUKEwjSenSyJHnAhWXTX0KHWw1BqoQFjAaegQIAhAB&usg=AOvVaw2ou89f5fahKqUBqZ gmLuIc&cshid=1579502355346.

VIDEO REFERENCES:

1. https://www.youtube.com/watch?v=5KLBrnauilg

Course Code: PGM-CHE-AC-C504

Course Title: Experiments in Analytical Chemistry Credits: 3 Duration: 72 Hours Maximum Marks: 100

Course Objectives:

1. To enable students in understanding the knowledge of separation and characterization

2. To enable students to carry out, record and analyze the result of analytical experiments

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Understand the quantitative approach towards various instruments
- CO2: Identify appropriate method to carry out quantitative analysis for desired samples

CO3: Perform titrimetric and spectrophotometric analysis

CO4: Develop good laboratory practices, both conceptually and practically

This course consists of seven units of experiments in various areas of Analytical Chemistry. Each Unit is equivalent to twelve hours duration. Any six units experiments to be performed.

UNIT I: Analysis of Pharmaceutical Tablets / Samples

- 1. Estimation of calcium from dietary supplements using Murexide indicator
- 2. Estimation of Ibuprofen / Paracetamol
- 3. Estimation of sulphadiazine / sulphonamide
- 4. Determination of neutralising power of tablets of different brands and compare effectiveness
- 5. Determination of iron using Zimmermann-Reinhardt reagent by titrating against potassium permanganate
- 6. Estimation of iron from given pharmaceutical drug sample using thioglycolic acid

UNIT II: Ion Exchange Chromatography and Solvent Extraction Method

- 1. To determine the capacity of a cation exchange resin
- 2. To determine the capacity of an anion exchange resin
- 3. To determine the Fe ion as Fe-oxine complex using Butyl acetate/ CHCl₃ asextracting solvent
- 4. To separate the acidic, basic and neutral compounds from the mixture by solvent extraction

UNIT III: Electrochemical Method

- 1. pH-metric determination of hydrolysis constant of aniline hydrochloride
- 2. pH-metric determination of the acid-base dissociation constant and isoelectric point of amino acid
- 3. pH metric determination of dissociation constant of dibasic, oxalic acid
- 4. Potentiometric estimation of carbonate and bicarbonate from the mixture
- 5. Potentiometric determination of dissociation constant for Cu-ammonia complex
- 6. To determine the critical micelle concentration of the detergent using conductometer

UNIT IV: Gas and HPLC Chromatographic Analysis

- 1. Optimum flow rate for the determination of chloroform using Van Deemter equation
- 2. Quantitative analysis of a mixture of chloroform and carbon tetrachloride
- 3. Gas chromatographic analysis for a mixture of gases like O₂, N₂ and CO₂
- 4. Analysis of mixture of alcohols using HPLC
- 5. To study the quantitative assay of ampicillin injection powder by using HPLC
- 6. To analyze the mixture of two hydrocarbons (Toluene and Nitrobenzene) by HPLC
- 7. Analysis of Ibuprofen/Paracetamol (analgesics) in a commercial sample/tablet byHPLC
- 8. To develop and validate the analytical method of any one drug using GC/HPLC
- 9. To determine the number of theoretical plates by HPLC using Acetophenone as reference material

UNIT V: Simple Chromatography

- 1. To separate alpha amino acids by paper chromatography
- 2. To separate the two organic compounds from the mixture by TLC
- 3. To separate the leaf pigments: chlorophyll 'a' chlorophyll 'b', carotene and xanthophylls by paper chromatography
- 4. To determine the Rf value of glycine by ascending paper chromatography
- 5. To separate sugars and amino acids by paper and thin layer chromatography
- 6. To separate the mixture of o- and p- nitro anilines by column chromatography
- 7. To study the presence of lactose in milk by descending paper chromatography

UNIT VI: Spectrophotometric Method

- 1. To determine pka value of methyl red indicator at room temperature
- 2. To determine the indicator constant and isobestic point of an indicator
- 3. To determine the stoichiometry and stability constant of ferric salicylic acid complexby Job's method and mole ratio method
- 4. To determine the amount of each p-nitrophenol and m-nitrophenol from the mixture by spectrophotometric titration using standard NaOH solution at $\lambda \max = 280 \text{ nm}$
- 5. To record the UV absorption spectrum of acetone in n-hexane and identify the various transitions
- 6. To estimate the amount of aspirin and caffeine from APC tablet by UV-Visible spectrophotometry
- 7. To study the iodination of acetone by spectrophotometric method
- 8. To estimate the amount of arsenic in dried shrimp by UV-Visible spectrophotometry using molybdenum blue method.
- 9. To estimate Pb/Hg by AAS method

UNIT VII: Interpretation Exercise

- 1. X-ray powder diffraction analysis of cubic compound:
 - a. Determination of Lattice constants and crystallite Size
 - b. Density
- 2. Interpretation of Mossbauer spectrum with reference to determination of: isomer shift; quadruple splitting; internal magnetic field; general comment
- 3. Interpretation of IR spectrum with reference to stretching vibration of: C=N; C=O; N-O; M-O
- 4. Interpretation of NMR spectrum with reference to calculation of chemical shifts and general comments
- 5. Interpretation of absorption spectra for:
 - a. Verification of the position of ligands in spectrochemical series
 - b. Calculation of spectral splitting parameters
 - c. Determination of geometry of a given compound (octahedral, tetrahedral, square planar)
- 6. Statistical revaluation of spectrophotometric data

REFERENCE BOOKS:

- 1. Vogel's Textbook of Quantitative Chemical Analysis, 6th Edition
- 2. Comprehensive Experimental Chemistry, V. K. Ahluwalia; New Age Publications
- 3. Experimental Physical Chemistry, F. Daniels and J. Williams
- 4. Experimental Physical Chemistry, R. C. Das and B. Behera
- 5. Practical Physical Chemistry, B. Viswanathan, P. S. Raghavan
- 6. An Introduction to Practical Biochemistry, D. T. Plummer; 3rd Edition, Tata McGraw-Hill, New Delhi
- 7. Advanced Physical Chemistry, J. B. Yadav; 14th Edition, Goel Publishing House
- 8. Systematic Experimental Physical Chemistry, S. W. Rajbhoj, T. K. Chondhekar; Anjali Publication, Aurangabad.

VIDEO REFERENCES:

1. https://www.youtube.com/watch?v=lha8dEBNFS4&t=251s

ELECTIVE COURSES

Course Code: PGM-CHE-AO-E501 Course Title: Advanced Mass Spectrometry Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

1. To enable students to understand the basics of Mass spectrometry, different ionisation methods, mass analysers and its application to solve structural problems of proteins

Course Outcome:

On successful completion of the course, the student will be able to:

- CO1: Choose a proper technique for the analysis of the desired compound
- CO2: Interpret and recognise various mass spectra
- CO3: Solve and elucidate structures of various organic compounds
- CO4: Explain and understand various ionisation processes and recognise different spectrabased on ionisation modes.

UNIT I: Ionisation Sources

Mass Spectrometry- introduction, principle, instrumentation of mass spectrometer; electron

ionization; chemical ionization; proton transfer; adduct formation; charge-transfer chemical ionization; reagent gas; negative ion formation; desorption chemical ionization; field ionization; field desorption; fast atom bombardment and liquid secondary ion mass spectrometry; field desorption; plasma desorption; laser desorption; matrix-assisted laser desorption ionization MALDI; thermo spray; atmospheric pressure ionization, atmospheric pressure- photo ionization; electro spray ionization; thermal ionization source; spark source; glow discharge source; inductively coupled plasma source.

UNIT II: Mass Analysers and Applications of MS

12 Hours

Quadrupole analysers; ion guide and collision cell; MSⁿ, time-of-flight analysers; linear time-offlight mass spectrometer; delayed pulsed extraction; reflectrons; tandem mass spectrometry with time-of-flight analyser; magnetic and electromagnetic analysers; tandem mass spectrometry in electromagnetic analysers; fragmentation reactions; tandem massanalysis, proteomics.

TEXT BOOK:

Mass Spectrometry: Principles and application, Edmond D. Hoffmann; Vincent Stroobant John Wiley

REFERENCE BOOKS:

- 1. Mass Spectrometry: A Textbook, Jurgen H. Gross; 2nd Edition, Springer
- 2. Electrospray and MALDI Mass Spectrometry: Fundamental, Instrumentations and Practicalities and Biological Applications, Richard B. Cole; John Wiley

WEB REFERENCES:

- 1. http://chemistry.emory.edu/msc/tutorial/mass-spectrometry-ionization.html
- 2. http://chemguide.co.uk/analysis/masspec/fragment.html
- 3. https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Map%3A_Organic_Chemi

 $(Wade)/11\%3A_Infrared_Spectroscopy_and_Mass_Spectrometry/11.0\underline{8\%3A_Fragm}entation_Patterns_in_Mass_Spectrometry$

4. https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Supplemental_Modules_(An alytical_Chemistry)/Instrumental_Analysis/Mass_Spectrometry/Mass_Spectrometers_(Instrumentation)/Mass_Analyzers_(Mass_Spectrometry)

5. Schwartz J C, Senko M W, Syka J E P. A two-dimensional quadrupole ion trap mass spectrometer. Journal of the American Society for Mass Spectrometry, 2002, 13(6): 659-669

Course Code: PGM-CHE-AO-E502

Course Title: Advanced NMR Spectroscopy Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

- 1. To enable students to understand basic aspects of nuclear magnetic resonance spectroscopy
- 2. To enable students to understand one-dimensional NMR, Chemical shifts, J-coupling, Interpretation of 1D NMR spectrum, basics of 2D NMR, different 2D NMR experiments and their application/interpretation

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Explain the concept of nuclear magnetic resonance spectroscopy
- CO2: Apply nuclear magnetic resonance spectroscopy for identifying organic compound
- CO3: Explain the concept of 2D NMR spectroscopy

CO4: Apply 2D NMR to identify organic compounds

11

UNIT I: NMR and ¹³C-NMR Spectroscopy

Nuclear magnetic resonance- theory, quantum description; classical description of NMR; types of NMR spectra; applications of proton NMR in qualitative and quantitative analysis (in general); CW and PFT techniques; Types of CMR spectra-undecoupled- proton decoupled-off-resonance decoupled (SFORD)-selectivity decoupled and gated ¹³CJ) and heteronuclear (¹³C-¹H, ¹³C-²HJ) couplings, nuclear overhauser effect, ATP (attached proton test), DEPT

UNIT II: 2D-NMR Spectroscopy

12 Hours Classification of 2D experiments- 2DJ resolved spectroscopy- HOMO and HETERO- 2DJ Resolved Spectra: correlation spectroscopy (COSY) - HOMO-COSY, 2D-INADEQUATE and NOESY.

TEXT BOOK:

Spectroscopic Identification of Organic Compounds, R. M. Silverstein, G. C. Bassler and T. M. Morril

REFERENCE BOOKS:

- 1. Spectroscopic Identification of Organic Compounds, R. M. Silverstein and Webster
- 2. NMR in Chemistry- A Multinuclear Introduction, William Kemp
- 3. ¹³C NMR for Organic Chemists, G. C. Levy, G. L. Nelson
- 4. Understanding NMR Spectroscopy, James Keeler; 2nd Edition

WEB REFERENCES:

- 1. http://chem.ch.huji.ac.il/nmr/techniques/2d/2d.html
- 2. http://www.cryst.bbk.ac.uk/PPS2/projects/schirra/html/2dnmr.htm
- 3. http://chem.ch.huji.ac.il/nmr/techniques/2d/cosy/cosy.html
- 4. http://www-keeler.ch.cam.ac.uk/lectures/2d_a4.pdf
- 5. http://chem.ch.huji.ac.il/nmr/techniques/2d/noesy/noesy.html

Course Code: PGM-CHE-AO-E503

Course Title: Applied Analytical Chemistry Credits: 2 **Duration: 24 Hours Maximum Marks: 50**

Course Objectives:

- 1. To enable students with a knowledge of various methods for identification of compounds using spectroscopy and to determine the quantitative analysis of sample
- 2. To enable students to analyse various constituents in compounds in various fields like clinical chemistry and cosmetics analysis

Course Outcomes:

On successful completion of the course, the student will be able to:

CO1: Understand the basic concepts used in clinical chemistry

CO2: Understand the chemistry of food and will be able to analyse different components in it

CO3: Get an idea about the nutritional values of food stuff

CO4: Apply the knowledge of analytical chemistry for analysing cosmetics

UNIT I: Food Analysis, Processing and Preservation

Food legislation and public health; nutritional value of foods; general methods for determination of moisture, ash, crude protein, fat, crude fibre, carbohydrates; analysis of foodand beverages (soft drinks, alcoholic drinks)-calcium, potassium, sodium and phosphates; edible oils and fats- general composition of edible oils; detection of purity; test for common edible oils; rancidity of fats and oils; estimation of rancidity; determination of ratio between saturated and unsaturated fatty acids; determination of- total fatty acid, acid value, iodine value, ester value, acetyl value, titre value,

peroxide value, R. M. value, P.V. value.Food processing and preservation-introduction; food processing; food preservation methods- freezing, drying, pasteurization, sterilization, irradiation, canning, concentration; analysis of preservatives; types of packing materials and properties; industrial requirements.

UNIT II: Clinical Chemistry and Cosmetics Analysis

12 Hours

A. Clinical Chemistry- introduction; sample collection and preservation of physiological fluids; composition of body fluid; detection of abnormal levels of certain constituents leading to diagnosis of diseases; analysis of physiological fluids like- blood, urine, serum; estimation of blood glucose, cholesterol, urea, haemoglobin, billirubin, uric acid, urea in urine, calcium, phosphate; physiological and nutritional significance of water soluble and fat-soluble vitamins, minerals.

B. Cosmetics analysis- introduction, analysis of lipsticks- determination of non-volatile matter, lakes and fillers; analysis of deodorants and antiperspirants for- Al, Zn, boric acid, chloride, sulphate, urea; analysis of face powder- fats, fatty acid, Ca, Mg, BaSO₄, Ti and Fe; oxides of Ti, Fe and Al (total); analysis of hair tonic: 2, 5- diamino toluene, KBrO₃, resorcinol, salicylic acid.

TEXT BOOK:

Food Composition and Analysis, H. O. Triebold, L. W. Aurand

REFERENCE BOOKS:

- 1. Introduction to Food Science and Technology Series, G. F. Stewart, M. A. America; Academic Press
- 2. Food Chemistry, H. K. Chopra, P. S. Panesar; Narosa Publication
- 3. Food Chemistry; Alex V. Ramani; M. J. P. Publishers
- 4. Analytical Chemistry of Foods, Ceiwyn S. James; 1st Edition, Blackie Academic and Professional Chapman and Hill Publisher
- 5. Chemical Analysis of Food, Pearson
- 6. Practical Biochemistry in Clinical Medicine, R. L. Nath; 2nd Edition, AcademicPublishers
- 7. Analytical Biochemistry, D. J. Holme, H. Peck; Longman
- 8. Bio analytical Chemistry, S. R. Mikkelsen, E. Corton; John Wiley and Sons
- 9. Chemical Analysis of Food and Food Products, H. B. Jacob; Van Westrand Reinhold

WEB REFERENCES:

- 1. https://www.news-medical.net/life-sciences/Clinical-Chemistry-Tests.aspx
- 2. https://www.sciencedirect.com/science/article/pii/0379073881901730
- 3. https://www.slideshare.net/mobile/parth241989/analysis-of-cosmetics-112070804018
- 4. https://www.sciencedirect.com/science/article/pii/S2214750015000165
- 5. https://sciencemonk.com/forensic-chemistry/

Course Code: PGM-CHE-AO-E504 Course Title: Bio analytical Chemistry Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

- 1. To enable students to the techniques routinely used in bio analytical laboratories
- 2. To enable students to study various bio analytical techniques used for diagnosis of diseases

Course Outcome:

On successful completion of the course, the student will be able to:CO1: Have valuable training in forensic science and biotechnology

CO2: Understand antibody-antigen interactions

CO3: Understand various bio analytical techniques used for medical diagnosis and research CO4: Understand the working of various biosensors used to detect biological compounds

UNIT I: Antibodies, spectroscopic methods for matrix characterization 12 Hours

Antibodies- Introduction, structural, functional properties of antibodies, polyclonal and monoclonal antibodies; antibody- antigen interactions; analytical applications of secondary antibody-antigen interactions: agglutination reactions and precipitation reactions; keys to immunochemical measurements; analytical applications of biological tracers; principle and applications of radioimmunoassay (RIA); enzyme linked immune sorbent assay (ELISA); Introduction to the concept of RTPCR; immuno histochemistry- important diagnostic tool; introduction to protein; method for total protein- Lowry, Smith, Bradford; proteinguantification methods; methods for total DNA- fluorometric, diphenylamine; total RNA; determination of total carbohydrate- ferricyanide, phenol sulphuric acid; Purpald assay for bacterial polysaccharides; free fatty acids.

UNIT II: Biosensors and Bio analytical approaches

Introduction to biosensors; examples of biosensor; configurations; response of enzyme-based biosensors; ferrocene-mediated amperometric glucose sensor; potentiometric biosensor for phenyl acetate; potentiometric immune sensor for digoxin; evanescent-wave fluorescence biosensor for bungaro toxin; optical biosensor for glucose based on fluorescence energy transfer; piezoelectric sensor for nucleic acid detection, enzyme thermistors; clinical genomics; proteomics and metabolomics; clinical diagnosis and screening; research and development; emerging pharmaceutical products, future perspectives; structure and characteristics of key transition metals, importance of transition metals in physiological processes, transition metals as mediators of disease processes, therapeutic implications of transition metals, determination of transition metals in nature.

TEXT BOOK:

Understanding Bio analytical Chemistry, V. A. Gault; John-Wiley and Sons

REFERENCE BOOK:

- 1. Analytical Biochemistry, D. J. Holme; Pearson Education Ltd.
- 2. The Principels of ion-selective electrodes and membrane transport, W. E. Morf
- 3. Bio analytical Chemistry, S. R. Mikkelsen; John-Wiley and Sons

WEB REFERENCES:

- 1. https://www.elprocus.com/what-is-a-biosensor-types-of-biosensors-and-applications/
- 2. Mehrotra, P. (2016, January 6). Biosensors and their applications A review. Journal of Oral Biology and Craniofacial Research. doi:10.1016/j.jobcr.2015.12.002
- 3. https://www.radiologyinfo.org/en/info.cfm?pg=bodymr
- 4. https://www.iaea.org/topics/radiotracers
- 5. https://www.antibodies-online.com/resources/17/1215/radioimmunoassay-ria/

Course Code: PGM-CHE-AO-E505 **Course Title: Calibrations and Validation**

Credits: 2 **Duration: 24 Hours Maximum Marks: 50**

Course Objectives:

- 1. To enable students to understand the validation characteristics of some procedures used in laboratory
- 2. To enable students to have an idea about ICH guidelines used in pharmaceutical industry

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Understand the qualification of laboratory equipment as a precondition of reliableanalytical testing
- CO2: Understand the basic rules of documentation in QA
- CO3: Calibrate the instruments of industrial importance
- CO4: Have the knowledge of ICH guidelines in method development

UNIT I: Regulations and Qualifications

Regulations: Regulatory requirements for analytical method validation; validation of analytical methods; complete method validation package, analytical data, protocol, plan, revisions and change controls; International Conference on Harmonization (ICH) Guideline Q2A: Validation of analytical procedures; linearity and range criteria and their role in instrumental method validation; GMP (US), Qualification: Overview of qualification of instruments; installation, operation and performance qualification (IQ, OQ, PQ) of analytical equipments; method validation for UV-Visible Spectrophotometer, IR Spectrophotometer, Spectrofluorometer, HPTLC, GC, HPLC; qualitative and quantitative method validation; parameters of validation; statistics in validation; detailed discussion on accuracy and precision role in method validation; protocols and interpretation.

UNIT II: Calibration

Calibration of analytical balance and pH meter; role of quantification limit and specificity; Limit of Detection (LOD) and Limit of Quantification (LOQ); Robustness and method validation; Ruggedness of chromatographic method; Ruggedness of sample preparation procedure; Calibration verses Qualification verses Validation; Case study for HPLC, UV; calibration of various instruments used for drug analysis like HPTLC, UV-Visible Spectrophotometer, IR Spectrophotometer, Spectro fluorimeter, GC, HPLC.

REFERENCE BOOKS:

- 1. The Theory and Practice of Industrial Pharmacy Lachman Edition
- 2. Web Resources in Pharmacy, In Pharma Publication, Bangalore
- 3. Schedule M
- 4. WHO Guideline
- 5. Analytical Method Development and Validation, Michael E. Swartz
- 6. Pharmaceutical Process Validation, Loftus and Nash
- Vogel's Textbook of Quantitative Chemical Analysis, J. Mendham, R. C. Denny, J. D. Banes, Thomas; 6th Edition, ELBS
- 8. Pharmaceutical Process Validation, Alfred H. Wachter
- 9. Validation and Qualification in Analytical Laboratories, Ludwig Huber; 2nd Edition, Wiley Publisher

WEB REFERENCE:

- 1. https://uc.xyz/1mhmZR?pub=link
- 2. https://www.researchgate.net/publication/8508200_Qualification_of_analytical_instr uments_for_use_in_the_pharmaceutical_industry_A_scientific_approach/link/02bfe5 0f872c59f953000000/download
- 3. https://www.slideshare.net/mobile/dhavalrock24/concept-of-ursdqiqoqpq
- 4. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4670047/
- 5. https://www.pharmaguideline.com/2010/05/calibration-of-uv-visible.html?m=1
- 6. https://nvlpubs.nist.gov

12 Hours

Course Code: PGM-CHE-AO-E506 Course Title: Chemometrics Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

To provide students with a basic tool in solving problems

Course Outcomes:

On successful completion of the course, the student will be able to: CO1: Handle computers and data sheet CO2: Handle statistical arrangements of data

UNIT I: Introduction to Data and Statistics

Introduction; univariate statistics review; probability; variance and sampling, linear regression and calibration data, digitization, and the Nyquist Theorem, detection limit, S/N ratio, and signal filtering; review of linear algebra: scalars, vectors, and matrices, matrix notation and matrix operations orthogonality, analysis of variance (ANOVA)- 1 variable, analysis of variance- 2 variables; introduction to MatlabTM: programmed, basics and layout, matrix operations in MatlabTM the diary command and examples, ANOVA in MatlabTM experimental design: factorial design, simple versus complex models, factorial design in MatlabTM; half-factorial design.

UNIT II: Multivariate Methods

A. Introduction to various multivariate methods; the six habits of a chemometrician; principle component analysis (PCA); data pretreatment- mean centering and normalization; PCA in MatlabTM.

B. Classical least squares (CLS), CLS in MatlabTM, inverse least squares (ILS).

C. Multiple linear regression (MLR); principle component regression (PCR); partial least squares, examples in MatlabTM; summary of multivariate methods; pattern recognition- supervised versus unsupervised pattern recognition, K nearest neighbours (KNN); soft independent modelling for chemical analysis (SIMCA), summary of pattern recognition.

TEXT BOOK:

Chemometrics, A Practical Guide; Kenneth R. Beebe, Randy J. Pell, and Mary BethSeasholtz, John Wiley and Sons, Inc., New York

REFERENCE BOOK:

The computer program MATLABTM will be required for some portions of the course

Course Code: PGM-CHE-AO-E507 Course Title: Techniques in Chemical Analysis Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

- 1. To illustrate to students the basic principles of modern instrumental methods
- 2. To enable students in understanding the basic theory underlying the construction of several common instruments and to become familiar with the operation of spectroscopic as well asnon-spectroscopic techniques

Course Outcomes:

On successful completion of the course, the student will be able to:

CO1: Develop an understanding of the range and theories of instrumental methods available in

12 Hours

analytical chemistry

- CO2: Have theoretical knowledge on selected instrumental methods of analysis
- CO3: Have the knowledge on analytical study by spectroscopy
- CO4: Understand and compare a range of analytical techniques and explain the underlying principles

UNIT I: Spectrophotometric, Nephelometry and Turbidimetry Analysis 12 Hours

Spectrophotometry- introduction; law of absorption; absorbance and transmittance spectrum; technique for colour comparison; radiation sources; wavelength selection; standard cells; photo detectors; spectrophotometer instrumentation- single and double beam spectrophotometer; presentation of spectral data; spectrophotometric titrations; applications- determination of Mn (II), Fe (III) as thiocyanate, Cu (II) using salicyladoxime, simultaneous determination of Mn (II) and Cr (VI).

Turbidimetry- introduction; principle; instrumentation; concentration effects; particle size and wavelength on scattering; turbidimetric titrations; applications.

Nephelometry- introduction; principle; instrumentation; concentration effects; particle size and wavelength on scattering; choice between the nephelometry and turbidimetry, turbidimetry and colorimetry, nephelometry and fluorimetry; applications.

UNIT II: Refractometry and Polarimetry

12 Hours

Refractometry- introduction; theory and measurement of refractive index, factors affecting refractive index; instrumentation; types of refractometers; molecular refractivity and chemical constitution; applications of refractometry- qualitative and quantitative analysis; plain curves; cotton effect curves.

Polarimetry- introduction, theory, principle; plane polarized light; optical activity; theory of optical activity; applications of optical activity; instrumentation; application of optical rotation method in rate constant determination; acid- inversion of cane sugar; relative strengths of acids; optical rotatory dispersion and Circular Dichroism and its applications; selection rules; deduction of absolute configuration of molecules; octant rule for ketones and cotton effect.

REFERENCE BOOKS:

- 1. Textbook of Quantitative Inorganic Analysis, A. I. Vogel, Longman
- 2. Instrumental Methods of Chemical Analysis, G. W. Ewing, McGraw-Hill
- 3. Basic Concepts of Analytical Chemistry, S. M. Khopkar
- 4. The Principals of ion-selective electrodes and membrane transport, W. E. Morf
- 5. Analytical Chemistry, G. D. Christian; 5th Edition, John Wiley and Sons, NY
- 6. Instrumental Methods of Chemical Analysis, H. Kaur, Pragati Prakashan
- 7. Instrumental Methods of Chemical Analysis, B. K. Sharma; Goel Publishing House

WEB REFERENCES:

- 1. http://rxpharmaworld.blogspot.com/2016/12/nephelometry-and-turbidimetry.html
- 2. http://www.environmentalpollution.in/pollution/regulation- andmonitoring/nephelometryand-turbidimetry-principle-theory-and-techniques/1880
- 3. https://www.microscopyu.com/techniques/polarizedlight/introduction-to-polarized-light

Course Code: PGM-CHE-AO-E508 Course Title: Thermal Methods of Analysis Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

1. To provide students with knowledge of thermal analysis to enable them to understand the principle of operation.

- 2. Obtaining basic knowledge on thermoanalytical methods
- 3. Application of thermoanalytical instruments in practice

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Choose the experimental conditions for the measurements and combine different thermoanalytical techniques
- CO2: Analyze and present the results of the measurements.
- CO3: Understand the principles of thermoanalytical techniques and combine different thermoanalytical techniques

CO4: Apply theoretical knowledge for practical analysis CO5: Analyse and present the results of the measurements

UNIT I: Thermogravimetric Analysis and Differential Thermal Analysis 12 Hours

Thermogravimetric Analysis- introduction; definition; instrumentation (all components to be

discussed); information from TGA curve; factors affecting TGA curves- instrumental, characteristics of sample; advantages and limitation of TGA; calculation of compound

composition, percent decomposition; applications of thermogravimetry; Derivative Thermogravimetry (DTG)- definition, comparison between TG and DTG.

Differential Thermal Analysis- introduction, definition; theoretical basis of DTA; DTA instrumentation (all components to be discussed); factors affecting the DTA curve; advantages and disadvantages of DTA; applications of DTA.

UNIT II: Differential Scanning Calorimetry and Thermometric Titrations 12 Hours

Differential Scanning Calorimetry- definition; instrumentation of DSC, types, factors affecting DSC curves; comparison between DTA and DSC techniques; applications.

Thermometric Titrations- introduction; definition; instrumentation (all components to be discuss); Numerical based on TGA and DTA curves to calculate percent loss and fix the formula of the sample are to be solved.

TEXT BOOK:

Fundamentals of Analytical Chemistry, D. A. Skoog, D. M. West, F. J. Holler, S. R.Crouch; 8th Edition

REFERENCE BOOKS:

- 1. Principles and Practice of Analytical Chemistry, F. W. Fifield, D. Kealy; Backwell Science Ltd., London
- 2. Vogel's Textbook of Quantitative Chemical Analysis; 6th Edition
- 3. Analytical Chemistry, G. D. Christian; 5th Edition, John Wiley, NY
- 4. Instrumental Methods of Chemical Analysis, H. Kaur; Pragati Prakashan
- 5. Instrumental Methods of Chemical Analysis, Chatwal and Anand, Himalaya Publishing House

WEB REFERENCES:

- 1. https://www.currenta.com/analytics/methods/thermoanalytical-methods.html
- http://web.abo.fi/instut/biofuelsGS-2/kursen/%C5A/lectures/Lectrure_Thermal%20Analysis.pdf
- 3. https://www.chemie-biologie.uni-siegen.de/ac/be/lehre/ss11/uebungensolidstate/summary_of_tg_and_dta.pdf
- 4. https://www.pslc.ws/macrog/dsc.htm
- 5. https://www.brainkart.com/article/Thermometric-Titrations-(TT)_30858/

Course Code: PGM-CHE-AO-E509 Course Title: Quality Assurance and Quality Control in Analytical Chemistry Credits: 2 Duration: 24 Hours Maximum Marks: 50

Course Objectives:

- 1. To enable students to understand the basics of quality control and quality assurance
- 2. To enable students to describe the types of packaging and regulatory aspects in food and pharmaceutical industries

Course Outcomes:

On successful completion of the course, the student will be able to:

- CO1: Explain the basics of quality assurance and quality control
- CO2: Know the types of packaging and regulatory aspects in food and pharmaceuticalindustries
- CO3: Handle reagents and chemicals appropriately
- CO4: Evaluate the quality assurance data

UNIT I: Introduction to Quality Assurance and Quality Control

Introduction to basic concepts, quality assurance; aspect of specification and tolerance; quality acceptance; sampling reality; cost aspect of quality decisions; quality control in raw materials; finished product; laws related to quality control; case studies of quality control in various industries like pharmaceuticals, agrochemicals, petrochemicals, dyes, plastics, polymers; safety in laboratory; importance of laboratory note book; cleaning and marking laboratory ware; measuring volume; calibrating volumetric flask; selecting and handlingreagents and chemicals; methods of quality assessment- internal and external; evaluating quality assurance data- prescriptive approach and performance based approach.

12 Hours

UNIT II: Standard Method, Analysis; Packaging and Regulatory Aspects 12 Hours A. Development of a standard method and analysis- introduction; optimising experimental procedure (Standard Operating Procedures); verifying the method- single-operator characteristics; blind analysis of standard samples; ruggedness testing; validating standard method; two-sample collaborative testing and analysis of variance.

B. Packaging and Regulatory Aspects- introduction; types of packing material and regulations; acts in food and pharmaceutical industries; testing of material for packing; legal aspects in packing; regulatory aspects of foods, drugs and cosmetics; food safety and Standards Act, 2006; I.S.I., AGMARK, other standards for foods and cosmetics with reference to testing of foods, drugs and cosmetics; raw material testing; Government authorities concerned with testing- their qualification, duties, powers, procedure to follow; records to be maintained under the Acts; C.G.M.P. and C.G.L.P.S.; Department of WHO certification.

TEXT BOOK:

Fundamentals of Analytical Chemistry, D. A. Skoog, D. M. West, F. J. Holler, S. R.Crouch; 8th Edition

REFERENCE BOOKS:

- 1. Quality Assurance in Analytical Chemistry, W. Funk, V. Dammann, G. Donnevert; VCH Weinheim
- 2. Principles and Practice of Analytical Chemistry, F. W. Fifield, D. Kealy; Backwell Science Ltd. London
- 3. Vogel's Textbook of Quantitative Chemical Analysis; 6th Edition
- 4. Modern Analytical Chemistry, D. Harvey; McGraw-Hill Education
- 5. Analytical Chemistry, G. D. Christian; 5th Edition, John Wiley and Sons, NY
- 6. Instrumental Methods of Chemical Analysis, H. Kaur; Pragati Prakashan

- 7. Pharmacopeia of India, Volume I and II
- 8. Quality in the Analytical Chemistry Laboratory, E. Prichard; John Wiley
- 9. Principals of Package Development, Gribbinetal
- 10. Modern Packaging Encyclopaedia and Planning Guide- MacqraWreyco
- 11. Government of India Publications of Food Drug Cosmetic Acts and Rules

WEB REFERENCES:

- 1. https://asq.org/quality-resources/quality-assurance-vs-control
- 2. https://nvlpubs.nist.gov/nistpubs/Legacy/IR/nbsir85-3105.pdf
- 3. https://www.who.int/water_sanitation_health/resourcesquality/wqmchap9.pdf
- 4. https://www.who.int/medicines/areas/quality_safety/quality_assurance/control/en/
- 5. https://www.who.int/tdr/publications/documents/glp-handbook.pdf

NOTE: Students can opt either PGM-CHE-AO-D508: Dissertation; or PGM-CHE-AO-M 509: Modules in Experimental Chemistry and PGM-CHE-AO-I510: Internship Modules; bothcourses together.

PGM-CHE-AO-D510: DISSERTATION

The dissertation must comprise of original research and may be conducted either at the Institute or with approval, in an outside institution or company e. g., the student's employers. The guiding teacher may serve some laboratory hours for industry work.

The dissertation work is to be submitted for: evaluation and Viva Voice examination at the end of Semester IV.

Written test must be conducted regarding the basic principles of techniques or instruments used with respect to the area of dissertation topic.

Students may be assessed based on designing the question bank by students on dissertation topic.

PGM-CHE-AO-M 511: MODULES IN EXPERIMENTAL CHEMISTRY 96 Hours

There are Seven Modules on experiments of various areas of Chemistry. Each Module is equivalent to eight hours duration per week.

INSTRUMENTATION MODULE I: IR SPECTROSCOPY

- 1. Quantification of acetyl group from polymers using IR
- 2. Plasticizer from PVC using IR
- 3. Determination of ethanol in gasoline
- 4. Spectral analysis of different compounds (synthesised inorganic complexes and organic compounds)
- 5. Following micro scale reaction using FTIR

MODULE II: POTENTIOMETRY

- 1. Potentiometric determination of reducing sugars
- 2. Potentiometric titration using graphite sensor
- 3. Kinetics of bromination reaction: A potentiometric study
- 4. Non-aqueous titration containing mixture of aniline and ethanolamine

MODULE III: GAS CHROMATOGRAPHY

- 1. Plasticizer from PVC using GC
- 2. Synthesis of high boiling organic compound by derivatisation and analyses by GC
- 3. Separation of alcoholic mixtures
- 4. Determination of alcoholic content in: i. Beer ii. Wine iii. Local drinks
- 5. Gas chromatographic analysis for: i. automobile exhaust ii. Cigarette smoke
- 6. Analysis of preservatives from solid and liquid samples (extraction, sample preparation and analysis)

MODULE IV: HPLC

- 1. Determination of caffeine content in: i. Tea ii. Coffee iii. Soft drinks iv. Chocolates
- 2. Qualitative and quantitative analysis of pharmaceutical Drug (e. g. aspirin)
- 3. Purity of the solvents using HPLC

MODULE V: TG/DTA/DSC

- 1. Determination of the purity of pharmaceuticals samples
- 2. Thermal properties of peanut proteins
- 3. Glass transition temperature of polymers (polymer to be used in preparation of membrane sensor)
- 4. Determination of water of crystallization in coordination compounds/ inorganic salts
- 5. Studies on thermal decomposition of Zinc NTA salt
- 6. DSC study on pharmaceutical product

MODULE VI: UV-VISIBLE SPECTROSCOPY

- 1. Method development and validation for a drug by UV/Visible spectrophotometer
- 2. Photometer (to build a photometer using LED) a guided-inquiry experiment to introduce analytical instrument
- 3. Determination of the composition of Ni and Fe (III) in a solution by spectrophotometric titration using Disodium salt of EDTA

MODULE VII: ATOMIC ABSORTION SPECTROSCOPY

- 1. Analysis of Na, K and Ca in water samples
- 2. Analysis of metal ion from soil /ore
- 3. Analysis of metal ion from alloys: Fe and Cr from steel / Zn from Brass / Sn andPb from solder
- 4. Analysis of Lead and cadmium in toys

MODULE VIII: NON-INSTRUMENTATION

- 1. Synthesis of nano composites
- 2. Analysis of Fats and oils- saponification value, iodine value, peroxide value, acid value
- 3. Determination of the rancidity of oil samples
- 4. Determination of acetic acid in vinegar
- 5. Synthesis of:
 - i. Polystyrene using suspension polymerisation
 - ii. Biginelli reaction (Solvent free synthesis)
 - iii. Hydrogenation using Pd-C
 - iv. Extraction of natural products using Soxhlet apparatus
- 6. Determination of copper in brass by complexometric method

PGM-CHE-AO-I512: INTERNSHIP MODULES

MODULE A: Internship at Industry; One week per Semester (Semester III and IV) 72 Hours MODULE B: Write up of the Internship work per Semester (Semester III and IV) 08 Hours MODULE C: Students to design two modules based on their experience at industry 16 Hours

96 Hours

*Assessment to be done at the end of Semester IV
