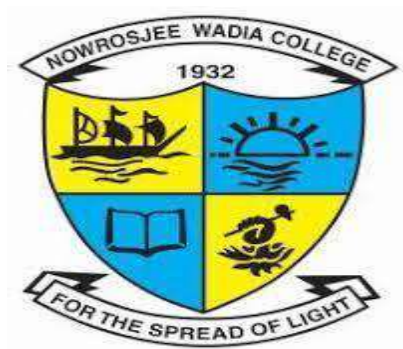


**Modern Education Society's**  
**Nowrosjee Wadia College**  
(An Autonomous College Affiliated To Savitribai  
Phule Pune University)



**Post-Graduate Program in Chemistry**

**New Syllabi (As Per National Education Policy-2020) for**

**M.Sc. Analytical Chemistry Part-II**

**To be implemented with effect from Academic Year 2024-2025**

## **1. Preamble:**

The global education development agenda reflected in the Goal 4 (SDG4) of the 2030 Agenda for Sustainable Development, adopted by India in 2015 - seeks to “ensure inclusive and equitable quality education and promote lifelong learning opportunities for all”. Such a towering goal will require the entire education system to be redesigned to support and foster learning, so that all of the critical targets for Sustainable Development can be achieved. National Education Policy 2020 is the first education policy of the 21<sup>st</sup> century and aims to address the many growing developmental imperatives of our country. This Policy proposes the revision and revamping of all aspects of the education structure, including its regulation and governance, to create a new system that is aligned with the aspirational goals of 21st century education, including SDG4. The NEP 2020 is based on the principle that education must develop critical thinking and problem solving abilities along with social, ethical, and emotional capacities.

The M.Sc. Chemistry syllabi are revised as per the guidelines of UGC, Government of Maharashtra and Savitribai Phule Pune University, Pune. With NEP-2020 in background, the revised curricula will articulate the spirit of the policy by emphasizing upon- integrated approach to learning; innovative pedagogies and assessment strategies; multidisciplinary and Interdisciplinary education; creative and critical thinking; student-centric participatory learning; imaginative abilities and flexible curricular structures to enable creative combination of disciplines for the study. The M.Sc. Chemistry Programme will transmit advanced knowledge of chemical sciences along with its fundamentals. In this programme, students will be empowered with assignments in academia and industry to provide the skills and information necessary for creating employment. The Programme exposes students to significant advances in chemical sciences as well as related fields through multidisciplinary and interdisciplinary courses. The design of the syllabi is such a way that it addresses chemical safety, green chemistry principles and industrial skills. It is intended to bring out the best in each student's ability, to sharpen their scientific temper, and to keep them up to date on recent developments in the field.

### **The Aims of the program are:**

- a) To impart basic and advanced knowledge of chemical sciences among students.
- b) To provide adequate blend of theory, computation and hands-on experiments.
- c) To provide higher education, disciplinary and inter/multi-disciplinary research oriented knowledge to the students.

- d) To provide a learned, skilled and creative pool of graduates who are ready to take up challenging assignments in different kinds of chemical industries, research institutions and academia.
- e) To foster responsible, proactive individuals who are equipped with rational thinking and competencies to address local challenges.

The M.Sc. Chemistry course structure consists of a well-balanced mix of Major Core, Major Electives, Research oriented courses, On-Job training/Internship and Project based learning. Out of total of 88 credits, 18 credits have been allotted to Research methodology and Project based learning. For M.Sc. Chemistry Degree, a student has to earn the minimum 88 credits from their four semesters. If students complete 44 credits in PG first year, he/she can exit with PG Diploma or continue with PG second year. The M.Sc. Chemistry course structure is based on following credit framework as per the guidelines of the university and government of Maharashtra.

#### Credit Framework for M.Sc. Chemistry Programme

Level	Semester	Credits Related to Major		Research Methodology (RM)	Internship Job Training (OJT)	Research Project (RP)	Total
6.0	I	10 (T) + 4 (P)	2 (T) + 2 (T/P)	4	0	0	22
	II	10 (T) + 4 (P)	2 (T) + 2 (T/P)	0	4	0	22
<b>Exit Option:</b> Award of PG Diploma on Completion of 44 credits at 6.0 level (PG First Year) or Continue with PG Second Year							
6.5	III	10 (T) + 4 (P)	2 (T) + 2 (T/P)	0	0	4	22
	IV	8 (T) + 4 (P)	2 (T) + 2 (T/P)	0	0	6	22
<b>Total 2 Years</b>		<b>54</b>	<b>16</b>	<b>4</b>	<b>4</b>	<b>10</b>	<b>88</b>
<b>Abbreviation:</b> T – Theory, P - Practical							

#### Guidelines for conduction of classes:

- a) A student has to attend 1-hour classroom teaching per week for one credit of theory and 2 hours' lab work/problem-solving session/ related activities per week for one credit of practical. Practical sessions (lab work/problem-solving session/related activity) will be conducted in batches. A batch for such sessions will be of size maximum of 08 students.

- b) 4 Credit courses will have 60 lectures (48 L + 12 T) and 2 Credit courses will have 30 lectures (24 L + 6 T)
- c) The Department may conduct necessary lectures/workshops as a part of OJT.
- d) Each course of 4 credits will carry 100 marks and 2 credit courses will carry 50 marks.
- e) There will be Continuous Internal Evaluation (CIE) and Semester End Examination (SEE) for each course.

**Evaluation process:**

- a) The CIE will be based on minimum two internal tests (IT). In addition, a teacher may consider one or more of the following. (i) Home Assignments (ii) Seminar/Presentation (iii) Laboratory assignment (iv) Group Discussions (v) Oral (vi) Research Paper/Book Review (vi) Technology Demonstration (vi) Case study (vii) Survey report, etc.
- b) Students has to score a minimum of 40 % separately in CIE and SEE, otherwise the result of such a course will be FAIL.

## 2. Programme Outcomes (POs)

<b>PO No.</b>	<b>PO Statement</b> After completing the Master of Science degree students are able to	<b>Knowledge and Skill</b>
<b>PO-1</b>	Learn the terms, theories, assumptions, methods, principles, theorem statements and classification	Disciplinary knowledge
<b>PO-2</b>	Fix out the problem and resolve it using theories and practical knowledge.	Critical thinking and Problem solving
<b>PO-3</b>	Inculcate his knowledge for carrying projects and advanced research related skills.	Research related skill
<b>PO-4</b>	Actively participate in team on case studies and field-based situations.	Cooperation/Team work
<b>PO-5</b>	Analyze and interpret ideas, evidences and experiences with learned scientific reasoning	Scientific reasoning
<b>PO-6</b>	Aware and implement the subject facts that can be applied for the personal and social development	Reflective thinking
<b>PO-7</b>	Use digital literacy to retrieve and evaluate subject related information	Information/Digitally literacy
<b>PO-8</b>	Get moral and ethical values for society as well as in research	Moral and ethical awareness
<b>PO-9</b>	Give analytical reasoning to interpret research data.	Analytical Reasoning
<b>PO-10</b>	Improve their managerial skills and abilities in subject related activities.	Leadership readiness/qualities
<b>PO-11</b>	Inculcate continuous learning habit through all available resources.	Lifelong readiness/qualities

### 3. Programme Specific Outcomes (PSOs)

<b>PO-No.</b>	<b>Outcomes</b>	<b>Component</b>
<b>PSO-1</b>	Demonstrate a comprehensive knowledge of all disciplines.	Disciplinary knowledge
<b>PSO-2</b>	To assess and evaluate facts, claims and arguments using their scientific knowledge	Critical thinking
<b>PSO-3</b>	To define a problem, analyse, interpret and draw conclusion by planning, implementing and reporting the results of an experiment.	Research-related skills
<b>PSO-4</b>	To access, evaluate and apply a variety of useful sources	Information/digital literacy
<b>PSO-5</b>	To participate in multicultural society and communicate the subject knowledge for the betterment of society	Multicultural competence
<b>PSO-6</b>	To acquire knowledge and skills including “Learning how to learn” that are necessary in learning activities throughout life	Lifelong learning

#### 4. Course Structure

### M. Sc. Analytical Chemistry Part-II

#### Semester III

Sr. No.	Course Title	Course Code	Major Core/ Major elective	Credits
1	Thermal and extraction techniques		Major Core	4T
2	Advanced chromatographic method and their applications		Major Core	4T
3	Applied electroanalytical techniques		Major Core	2T
4	Applied Physical Chemistry Practical -III		Major Core	2P
5	Applied Organic Chemistry Practical-III		Major Core	2P
6	A. Soil, fertilizer and Water analysis OR B. Food Analysis OR C. Analytical method development and validation		Major elective	2T
7	A. Applied Inorganic Chemistry Practical-III (Soil, water and fertilizer analysis) OR Applied food analysis practical		Major elective	2P
8	Research Project		RP	4

## Semester IV

Sr. No.	Course Title	Course Code	Major Core/ Major elective	Credits
1	Advanced Analytical spectroscopic techniques		Major Core	4T
2	Chemical methods of pharmaceutical analysis		Major Core	4T
3	Applied Physical Chemistry Practical -IV		Major Core	2P
4	Applied Organic Chemistry Practical-IV		Major Core	2P
5	A. Analytical techniques of polymer, paints and pigments OR B. Laboratory Automation and Sensor Based Techniques OR C. Forensic analytical chemistry		Major elective	2T
6	A. Applied Inorganic Chemistry Practical-IV OR B. Applied analytical chemistry practical		Major elective	2P
7	Research Project		RP	6

## 5. Detailed Syllabus

### Semester III

#### Major Core Paper-1-Thermal and extraction techniques (4 Credits, 60 L)

##### Section-I: Thermal Methods of Analysis (2 Credits, 30 L T)

###### 1. Introduction to Thermal Methods [2 L]

Introduction, Definitions: Thermal analysis, A Thermal Diversion, General apparatus, Factors affecting thermal analysis results: The sample, The crucible, The rate of heating, The atmosphere, The mass of the sample, Simultaneous and complementary techniques.

(Ref. 1: 1-21)

###### 2. Thermogravimetry [6 L]

Introduction, Definition of thermogravimetry, Apparatus: The balance, Furnace, Programmer, Samples, Temperature calibration, Atmosphere, Measurement of  $\alpha$  and  $d\alpha/dt$ , Applications of Thermogravimetry: Thermogravimetric curve for Decomposition of Magnesium Hydroxide, Calcium oxalate monohydrate and Copper sulphate pentahydrate, Degradation of polymers, Analysis of mixtures: mixtures of alkaline earth oxalates, polymer blends, soils, Oxidation studies, Reduction studies, Controlled rate thermogravimetry and Hi-Res™ TGA, Polymer blends, Drugs.

(Ref. 1:22 to 62)

###### 3. Differential Thermal Analysis and Differential Scanning Calorimetry [8 L]

Introduction, Definitions: Differential thermal analysis (DTA), Differential scanning calorimetry (DSC), Apparatus: The sensors, The furnace and controller, The computer and display, The reference material, Theory of DTA and DSC, Heat flux DSC, Power-compensated DSC, The effect of higher temperatures, Sample size, Calibration, Applications: Physical changes and measurements (crystalline phase transitions, potassium nitrate, liquid crystalline transitions, thermoplastic polymer phase change in heat capacity measurements, glass transition temperatures), Chemical reactions, Inorganic compounds and complexes (calcium oxalate monohydrate, metal complexes, high alumina cements, clays and other minerals), Organic compounds (oxidative degradation, protein denaturation, polymer degradation).

(Ref. 1: 63-113).

#### **4. Thermo mechanical and Dynamic Mechanical Analysis [6 L]**

Introduction, Definitions: Thermo mechanical analysis, Dynamic mechanical analysis, Mechanical moduli, Thermo mechanical analysis: Apparatus(probes, calibration), Applications: coefficients of expansion, solvent swelling of polymers, phase transitions, sintering), Chemical reactions (inorganic hydrates, polymer cure), Dynamic Mechanical Analysis: Apparatus (DMA configurations, calibration) Applications: glass transition temperatures, beta and other transitions, relaxation kinetics, polymer miscibility, characterizing cross-linking, studying problem samples, characterizing film formation.

**(Ref. 1:** 123-151)

#### **5. Simultaneous Techniques and Product Analysis [5 L]**

Introduction, Simultaneous Thermal Analysis: Simultaneous TG-DTA and TG-DSC, Applications: Sodium tungstate dihydrate, fire-retarded wood, poly (vinyl chloride), pharmaceuticals, reactive atmosphere effects, Evolved gas analysis: Definition, Apparatus, Detection and identification of evolved gases: Physical methods, Chemical methods, Spectroscopic methods (mass spectrometry (MS) and simultaneous TG-MS) calcium oxalate monohydrate, poly (ethylene oxide), brick clays).

**(Ref. 1:** 163-175)

#### **6. Problem Solving and Applications of Thermal Methods [3 L]**

Introduction, List of examples, Problems: Inorganic materials, Polymeric materials, Fine chemicals and pharmaceuticals, Other materials, Solutions to problems.

**(Ref. 1:** 206-270) (This topic is for student's self-preparation.)

#### **References:**

1. Thermal Methods of analysis Principles, Applications and Problems, P. J. Haines, Springer-Science Business Media B.V. First Ed. (1995).
2. Principles of Thermal Analysis and Calorimetry, P. J. Haines, Royal Society of Chemistry.
3. Principles and Applications of Thermal Analysis, Paul Gabbott, Blackwell Publishing Ltd. (2008).
4. Thermal Analysis in Practice, Fundamental Aspects, Matthias Wagner, Hanser Publications, 2018.

## Learning Outcomes

At the end of the course, students will be able to:

1. Know the importance of thermogravimetric analytical techniques.
2. Explain instrumentation in thermogravimetry and extraction techniques.
3. Understand the basic principles of thermogravimetry and extraction techniques.
4. Differentiate among the various methods of thermogravimetry.
5. Explain /Describe the applications of electrochemistry and thermogravimetry in industries and in analytical laboratory.
6. Apply/select particular method of analysis for characterizing specific sample.
7. Solve numerical problems on thermogravimetry.
8. Interpret thermogram, differential thermogram and DSC thermogram.

### Section-II: Analytical Extraction Techniques (2 Credits, 30 L T)

#### 1. Pre and Post Extraction Consideration[2 L]

Organic compounds of interest, pre-sampling issues, sampling strategies-solid, aqueous and air samples, chromatographic method of analysis, sample preconcentration methods. (Ref-1: 1-29)

#### 2. Classical Approach for Aqueous Extraction [7 L]

Introduction, Liquid-Liquid extraction (LLE), Theory of LLE: distribution ratio and coefficient, solute remaining unextracted, percent extraction, separation factor, factors favouring solvent extraction, quantitative treatment to solvent extraction equilibria, synergic extraction, extraction reagents for metals, selection of solvents, solvent extraction, problems with LLE process), purge and trap for volatile organics in aqueous samples, Examples of Solvent Extraction- estimation individual metal ions Be, B, Cu, Fe and Pb by solvent extraction. Problems. (Ref-2: relevant pages and Ref.-1: 39-45)

#### 3. Solid Phase extraction (SPE) [7L]

Introduction, Types of SPE media, SPE formats and apparatus, method for SPE operation, solvent selection, factors affecting SPE, selected methods of analysis for SPE: application of normal phase SPE, application of reversed phase SPE, application of ion exchange SPE, applications of molecularly impaired polymers, Automation and On-Line SPE and its applications. (Ref-1: 49-78)

#### 4. Solid phase micro-extraction [7L]

Introduction, theoretical considerations, experimental, Methods of analysis: SPME-GC: direct immersion SPME, headspace SPME, analysis of compounds from solid matrix, other SPME-GC application. Methods of analysis: SPME-HPLC-MS: analysis of abitic dehydroabietic acid in food samples, analysis of fungicide in water. Automation of SPME and its application, New development in micro extraction (Introduction, stirbas sorptive extraction, liquid phase micro-extraction, , membrane micro extraction, micro extraction in packed syringe).(Ref-1: 85-110, Ref-3)

## **5. Solid -Liquid Extraction, Microwave extraction [7 L]**

**Classical Approach:** Introduction, Soxhlet extraction, Automated Soxhlet extraction, other approaches, **Pressurized Fluid Extraction:** Introduction, theoretical consideration, Instrumentation for PFE, method development and applications. **Microwave assisted extraction:** Introduction, instrumentation, Applications(**Ref-1:** 125-174)

### **References**

1. Extraction Techniques in Analytical Science, John R. Dean, Wiley
2. Vogel's Textbook of quantitative Chemical Analysis, sixth Ed., Mendham, Denney, Barnes, Thomas, Pub: Pearson Education.
3. Solid Phase Microextraction, A Practical Guide, Edited by Sue Ann Scheppers Wercinski, CRC press, Taylor and Francis.

### **Learning outcomes –**

At the end of course students should able to-

1. Define / understand various terms in analytical extraction and method development and validation.
2. Explain instrumentations and methodology in analytical extraction.
3. Explain / describe basic principles of analytical extraction method development and validation.
4. Explain /Describe applications analytical extraction and method development and validation in industry and in analytical laboratory.
5. Apply / select particular method of analysis for sample to be analysed.
6. Solve numerical problems on analytical extraction and method development and validation.
7. Develop analytical method for analysis of given sample. Apply statistical treatment to the analytical data. Select appropriate parameters for the development of analytical method
8. Differentiate among the methods of analytical extraction.

## **Major Core Paper-2-Advanced Chromatographic Methods of Analysis (4 Credits, 60 L)**

### **Section-I: Mass spectrometry and Gas Chromatography (2 Credits, 30 L T)**

#### **1. Mass Spectrometry [5 L]**

Fundamentals, Electron ionization, Chemical ionization, Instrumentation: Quadrupole mass spectrometers, Magnetic sector mass spectrometers, TOF mass analyser, detector; Interpretation of mass spectra, Types of ions Isotopic abundances and characteristic ion clusters, Nitrogen rule and rings-plus-double-bonds, steps in interpretation, Examples (**Ref-1:** 39-72, Supplementary Ref.- 4)

#### **2. Fundamentals of Chromatographic Methods of Analysis [4 L]**

Fundamentals of Chromatographic Separation (overview, the development of chromatogram), Characteristics value in chromatogram, Chromatographic theories (plate theory, kinetic theory),  $R_s$  as measure of peak separation, qualitative and quantitative analysis. Problems. (**Ref-2,** Supplementary Ref-1, 6)

#### **3. Gas Chromatography [6L]**

Retention data and partition coefficient, separation in the gas phase, Components of gas chromatography: Carrier gas, sample injection, split injection, splitless injection, cold on column injection, programmable temperature vaporization, head space injection, solvent effects, column, detectors- TCD, FID, ECD, Stationary phases for GC: stationary phases for packed column, capillary column, deactivation of surface, different stationary phases, Applications of GC, Problem on quantitative analysis. (**Ref.-2,** Supplementary Ref-1, 6)

#### **4. Gas Chromatography-Mass Spectrometry [10 L]**

Vacuum and gas flow, Basic principles, Analysis of vacuum and gas flow, Interfaces, Computerization, Computerized operation, Characteristics, Data analysis, Reconstructed gas chromatogram, Mass chromatogram, Selected ion monitoring, Background subtraction, Biller-Biemann stripping technique, Compound identification using reference spectra matching, Mass spectral compilations, Methods of computerized mass spectral search, Commercial mass spectral computer search systems, Quantitative analysis by selected ion monitoring, Choice of ions: basic considerations, Magnetic sector versus quadrupole analysers, Identification and quantitation procedures, Use of isotopically labelled standards, Precision, accuracy and limit of detection. (**Ref-1:** 79-134)

#### **5. Applications of GC and GC-MS [5 L]**

1. Quantitative analysis by GLC-different methods, Elemental Analysis using Gas Chromatography, analysis of Al, analysis of a mixture using the internal normalisation method, determination of sucrose as its trimethylsilyl derivative using gas-liquid chromatography, **Ref-4**

2. Phenols in waste water by LLE-GC method (sec-6420 phenols), Organochlorine pesticides in water: LLEGC method-1, LLEGC method-2 (sec-6630 organochlorine pesticides), volatile organic compounds – Purge and trap capillary column GC-MS method (Sec-6200-A,B,C), Tributyl tin by GC-MS and FID method (Sec-6710-A,B,C) **Ref- 5**

## References

1. Basic Gas Chromatography Mass Spectrometry, Principles and Techniques, F.W. Karasek and R.E. Clement, Elsevier, (Elsevier Science B.V.) 1988
2. Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley –VCH
4. Vogel's, Textbook of Quantitative Chemical Analysis 6th Ed.
5. Standard methods for the examination of water and waste water, 23rd Ed. Rodger Baird, Andrew Eatson, Eugene Rice, jointly published by: American Public Health Association, American Water Works Association, Water Environment Federation,
6. Forensic applications of Gas Chromatography by Michelle Carlin and John Dean, CRC press, 2013)

## Section-II: Liquid Chromatography (2 Credits, 30 L T)

### 1. Instrumentation of HPLC [5 L]

Introduction: HPLC-A powerful separation method, A first HPLC experiment, Liquid chromatographic separation modes, The HPLC instrument, Pumps: General requirements, The short-stroke piston pump, Preparation of Equipment up to Sample Injection: Selection of the mobile phase, Preparation of the mobile phase, Gradient systems, Sample injectors, Sample solution and sample volume; Solvent Properties: Table of organic solvents, Solvent selectivity, Miscibility, Buffers, Shelf life of mobile phases, The mixing cross; Detectors: General, UV detectors, Refractive index detectors, Fluorescence detectors, Electrochemical (amperometric) detectors, Light-scattering detectors, Multiple detection; Columns and Stationary Phases: Columns for HPLC, Precolumn, General properties of stationary phases, Silica, Chemically modified silica, Styrene-divinylbenzene, Column care and regeneration(**Ref-2:** 1-9, 59-136, Ref-1)

### 2. HPLC Methods [6 L]

- a) **Adsorption Chromatography:** Normal-Phase Chromatography: What is adsorption?, The eluotropic series, Selectivity properties of the mobile phase, Choice and optimization of the mobile phase, Applications(**Ref.-2:** 159-168, Ref-1)
- b) **Reversed-Phase Chromatography:** Principle, Mobile phases in reversed-phase chromatography, Solvent selectivity and strength, Stationary phases, Method development in reversed-phase chromatography, Applications, Hydrophobic interaction chromatography. (**Ref.-2:** 173-191, Ref-1)
- c) **Chromatography with Chemically Bonded Phases:** Introduction, Properties of some stationary

phases, Hydrophilic interaction chromatography, (**Ref.-2:** 195-200, Ref-1)

**d) Ion-Exchange Chromatography:** Introduction, Principle, Properties of ion exchangers, Influence of the mobile phase, Special possibilities of ion exchange, Practical hints, Applications (**Ref.-2:** 203-213, Ref-1)

**e) Ion-Pair Chromatography:** Introduction, Ion-pair chromatography in practice, Applications (**Ref.-2:** 217-221, Ref-1)

**f) Ion Chromatography:** Principle, Suppression techniques, Phase systems, Applications (**Ref.-2:** 225-230, Ref-1)

**g) Size-Exclusion Chromatography:** Principle, The calibration chromatogram, Molecular mass determination by means of size-exclusion chromatography, Coupled size-exclusion columns, Phase systems, Applications. (**Ref.-2:** 231-244, Ref-1)

**h) Affinity Chromatography:** Principle, Affinity chromatography as a special case of HPLC, Applications. (**Ref.-2:** 249-252)

### **3. Analytical HPLC [3 L]**

Qualitative analysis, Trace analysis, Quantitative analysis, Recovery, Peak-height and peak-area determination for quantitative analysis, Integration errors, The detection wavelength, Derivatization, Unexpected peaks: Ghost and system peaks. (**Ref.-2:** 285-308)

### **4. Separation of Enantiomers [3 L]**

Introduction, Chiral mobile phases, Chiral liquid stationary phases, Chiral solid stationary phases, Indirect separation of enantiomers. (**Ref.-2:** 333-345)

### **5. LCMS Interface and applications [10 L]**

**Interface Technology:** Introduction, Thermo-spray interface, The electron spray interface (mechanism of electron-spray ionization, sample types, the electro-spray spectrum, structural information from electro spray ionization), atmospheric pressure chemical ionization interface and the mechanism of atmospheric pressure chemical ionization. Data acquisition (identification, quantitation-selected ion monitoring), Processing of mass spectra (total ion current trace, qualitative analysis, quantitative analysis). **Applications:** Molecular weight determination of small molecules (Method Development for Structural Studies, The Use of Target-Compound Analysis and LC-MS-MS for the Identification of Drug Metabolites, The Use of High-Accuracy Mass Measurements in Combination with LC-MS for the Structure Determination of Drug Metabolites, The Use of Cone-Voltage Fragmentation in Conjunction with High-Accuracy Mass Measurements and LC-MS for Metabolite Identification, (**Ref-3:** 75, 94-123, 189-218)

### **Chapter-6: Super Critical Fluid Chromatography and Extraction [3L]**

Properties of supercritical fluid, Supercritical fluid chromatography: Instrumentation and operating variables, effect of pressure, stationary phases, mobile phases, detectors, comparison with other

types of chromatography, Applications, supercritical fluid extraction: Advantages of SFE, instrumentation, of line and on line extraction, applications. (Ref-4: 856-865, supplementary Ref-1)

## References

1. Analytical Chemistry, Ed. by Kellner, Mermet, Otto, Valcarcel, Widmer, Second Ed. Wiley – VCH
2. Practical High-Performance Liquid Chromatography, Veronika R. Meyer, Fifth Ed. John Wiley and Sons, Ltd.
3. Liquid Chromatography Mass Spectrometry: An Introduction by Bob Ardery, Publisher: Wiley India Pvt. Ltd. (2003). A book from series- Analytical techniques in the Science.
4. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.

## Learning Outcomes –

At the end of course students should be able to-

1. Define / understand various terms in chromatography (GC and HPLC) and mass spectroscopy.
2. Explain instrumentations in chromatography (GC and HPLC) and mass spectroscopy.
3. Explain / describe i) basic principles of chromatography (GC and HPLC) and mass spectroscopy. ii) Separation in GC / HPLC column. iii) Functioning and construction of GC / HPLC/ MS detectors.
4. Explain / Describe applications chromatography (GC and HPLC) in industry and in analytical laboratory.
5. Apply / select particular method / instrumental parameters for analysis for sample GC / HPLC.
6. Solve numerical problems on chromatography (GC and HPLC) and mass spectroscopy.
7. Integrate GC and HPLC chromatogram, Mass spectrum
8. Differentiate among the chromatography (GC and HPLC) methods of analysis.

## Major Core Paper-3-Applied electroanalytical techniques (2 Credits, 30L T)

### 1. Coulometry [10 L]

Current voltage relationship during an electrolysis, Operating cell at fixed applied potential, constant current electrolysis, Electrolysis at constant working electrode potential, Coulometric methods of analysis, Faradays laws of electrolysis, Instrumentations-Constant current and constant voltage instruments, potentiostatic coulometry-Instrumentation and applications, coulometric titrations - apparatus and applications, problems. (Ref.-1:696-712, Ref-2: relevant pages)

### 2. Voltammetry and Polarographic Methods of Analysis. [20 L]

a) **Polarography** (linear scan polarography): Polarographic principles, Instrumentation (different types of microelectrode such as dropping mercury electrode, the static drop mercury electrode,

rotating disc and ring disc electrode, cell for polarography, reference and counter electrode and circuit diagram), polarogram and polarographic currents, charging or capacitive current, role of supporting electrolyte, factors affecting on polarographic wave, Ilkovic Equation, advantages and disadvantages of DME, polarographic maxima and maxima suppressors, interference due to dissolved oxygen, Applications (qualitative analysis, quantitative analysis by calibration curve and standard addition methods), problems. (Ref-1: 716-723, Ref-2, Supplementary Ref. 3 and 4)

**b) Hydrodynamic Voltammetry:** Hydrodynamic voltammetry and applications of hydrodynamic voltammetry (voltammetric detectors in chromatography and flow injection analysis, Voltammetric oxygen sensors, amperometric titration. (Ref-1: 723-735)

**c) Cyclic Voltammetry:** Principle of cyclic Voltammetry, cyclic voltamogram of  $K_3[Fe(CN)_6]$  and parathion (Fundamental studies), determination of analytes using CV, criteria of reversibility of electrochemical reactions, quasi-reversible and irreversible processes (Ref-1: 735-742 Ref-2: Relevant pages, Supplementary Ref.-5: 27-68)

**d) Pulse Polarography:** different types of excitation signals in pulse polarography, Differential pulse polarography, square wave polarography, and Stripping method. Voltammetry with ultra-microelectrode, Applications. (Ref-1: 742-753 2, Supplementary Ref. 3 and 4)

### References

1. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.
2. Vogel's Text Book of quantitative analysis 6th Ed.
3. Introduction to Instrumental Analysis by R. D. Braun, Pharmamed Press.
4. Analytical Chemistry, A Modern Approach to Analytical Science, Ed. by R. Kellner, J. M. Mermet, O. Otto, M. Valcarcel, H. M. Widmer, Second Ed. Wiley –VCH
5. Cyclic Voltammetry, Simultaneous Analysis and Reaction Mechanism, David K Gosser, VCH, 1994

### Learning Outcomes:

At the end of course, students should able to-

1. Define various terms in electroanalytical chemistry.
2. Explain instrumentation in electroanalytical chemistry.
3. Describe basic principles of electroanalytical chemistry.
4. Explain /Describe applications of electroanalytical chemistry in industry and in analytical laboratory.
5. Apply / select particular method of analysis for sample to be analysed.

### Major Core Paper-4-Applied Physical Chemistry Practical –III

Applied Physical Chemistry Practical –III (2 Credits, 60L Any 12 Practicals)	
Sr. No.	Title with Content
I	<b>Table Work (Compulsory Practical)</b> 1. Table Work: Calculation of mean deviation, standard deviation, error, absolute error and elimination of data.

<b>II</b>	<b>Instrumental Practicals (Any Eleven Practicals)</b>
<b>1.</b>	<b>Spectrophotometry / Colorimetry Technique</b> 1. Estimation of aspirin from given tablet by spectrophotometry. 2. Determination of amount of each copper and Bismuth or Copper and Iron (III) from the given mixture by spectrophotometric titration using standard EDTA solution.
<b>2.</b>	<b>Conductometry Technique</b> 1. Determination of relative strength of acetic acid, chloroacetic acid and trichloroacetic acid through measuring their $K_a$ value by conductivity
<b>3.</b>	<b>Potentiometry Technique</b> 1. Determination of Strength of commercial phosphoric acid by potentiometric. 2. To determine chloride and iodide from given halide mixture by potentiometry.
<b>4.</b>	<b>Flame photometry Technique</b> 1. Determination of K from water sample by calibration method and its confirmation by standard addition method using flame photometry. 2. Determination of Na and K from given mixture by working curve method using flame photometry.
<b>5.</b>	<b>Photoflurometry Technique</b> 1. Analysis of Riboflavin from vitamin supplementary capsules / syrup / tablet sample by working curve method using Photoflurometry.
<b>6.</b>	<b>Polarimetry Technique</b> 1. Determination of glucose from saline sample by polarimetry.
<b>7.</b>	<b>pH metry Technique</b> 1. Determine aspirin in tablet conventional titration and by pH metric titration.
<b>8.</b>	<b>Turbidimetry Technique</b> 1. Estimation of $Cl^-$ from water or saline sample or food sample by calibration curve method using turbidimetry.
<b>9.</b>	<b>HPLC Technique</b> 1. Analysis of Paracetamol by HPLC <b>References</b> 1. Pharmacopeia of India. 2. Biochemical methods, Sadashivam and Manickem, New Age international Publication. 3. General Chemistry Experiments, by Elias, Universities Press. 4. Vogel's textbook of quantitative chemical analysis, Sixth Ed. Mendham, Denny Barnes, Thomas, Pearson education. 5. Experiments in chemistry by D. V. Jahagirdar (Himalaya publication). 6. An introduction to practical Biochemistry, Third Ed. by Plummer, Tata Mc-Grew Publishing Company. 7. Quantitative Inorganic Analysis including Elementary Instrumental Analysis by A.I. Vogel's, 3rd Ed. ELBS (1964).
<b>Aims and Objectives of the Course:</b>	
<b>Student should understand and learn -</b>	
1. Calculations of mean deviation, standard deviation, error, absolute error and elimination	

- of data.
2. Preparation of various low concentration solutions.
  3. Standardization and Operations of different instruments
  4. Analysis of different compounds using different instrumental techniques

### Learning Outcomes

After studying this course -

1. Prepare solutions of various concentrations (like molar, normal, ppm, percentage etc.) solution.
2. Prepare solutions of various concentrations (like molar, normal, ppm, percentage etc.) solution.
3. Calibrate and use colorimeter, uv-visible spectrophotometer, photofluorometer, nephelometer, flame photometer etc.
4. Perform experiments using above instruments.

## Major Core Paper-5-Applied Organic Chemistry Practicals –III

### Applied Organic Chemistry Practical's- III ( 2 Credits, 60 L , Any 12 Practical's)

Unit No.	Title with Content	No. of Practicals
<b>I</b>	<b>Table Work (Compulsory Practical)</b> Characterization of organic compounds by VU-Visible, IR, NMR, CMR and Mass spectroscopy of <b>any two</b> drug compounds: <ol style="list-style-type: none"> <li>1. Aspirin</li> <li>2. Paracetamol</li> <li>3. Sulfanilamide</li> <li>4. Benzocaine</li> <li>5. Methyl Salicylate</li> </ol> (Actual spectra must be given for analysis to the students)	<b>2</b>
<b>II</b>	<b>Preparation and estimation of drugs (Any three drugs)</b> <ol style="list-style-type: none"> <li>1. Aspirin</li> <li>2. Paracetamol</li> <li>3. Sulfanilamide</li> <li>4. Methyl Salicylate</li> <li>5. Ibuprofen</li> <li>6. Any other drug molecules</li> </ol>	<b>3</b>
<b>III</b>	<b>Specifications tests as per Indian Pharmacopeia for drug molecules (Any two drugs)</b> <ol style="list-style-type: none"> <li>1. Aspirin</li> <li>2. Paracetamol</li> <li>3. Caffeine</li> <li>4. Any other drug molecules</li> </ol> (Description, Solubility, Identification, loss on drying, Limit tests etc.)	<b>2</b>
<b>IV</b>	<b>Analysis of food material (Any five Practicals)</b> <ol style="list-style-type: none"> <li>1. Estimation of Caffeine from tea powder/tea leaves</li> <li>2. Estimation of Tannin from tea or Coffee by Folin-Danis method</li> <li>3. Estimation of HMF from honey sample</li> </ol>	<b>5</b>

4. Determination of saponification and acid value of oil/soap sample
5. To determine the amount of acetic acid of commercially available vinegar by Volumetric / Potentiometric titration
6. Estimation of glucose from Glucon-D or any other glucose sample by Fehling's method.
7. Estimation of Vitamin-C from lemon/ Tablet using Dichlorophenol-Indophenol dye by Volumetric method

**References:**

1. Vogel Textbook of Quantitative Chemical Analysis, 6<sup>th</sup> Edition
2. Lab. Manual: Manual of methods of analysis of Foods, Vegetables- Fruit and vegetable products
3. Indian Pharmacopeia Volume I, 7<sup>th</sup> Edition
4. Indian Pharmacopeia Volume II, 7<sup>th</sup> Edition
5. Indian Pharmacopeia Volume III, 7<sup>th</sup> Edition
6. Post Graduate Chemistry Practicals–S.S.Kelkar, H.N.Patel, S.P.Turakhiya,A.G.Gadre, Himalaya Publishing House.

**Aims and Objectives of the Course-**

Sr.No.	Aims and Objectives
Student should understand and learn -	
1	Spectroscopic data such as UV, IR and NMR of some drug molecules
2	Preparation of various drug molecule and their estimation methods
3	Tests for detection of drug molecules as per IP
4	Analysis of food materials from Caffeine, Tannin, HMF, Glucose, Vitamin C, etc.

**Learning Outcomes:**

Sr.No.	Learning Outcomes
After studying this course -	
1	Student will be able to carry out preparation of drugs
2	Student will be able to carry out different methods of estimation of drugs
3	Student will be able to analysis of UV, IR, NMR and CMR spectroscopic data of drugs
4	Student will be able to estimate the various food samples in laboratory

**Major Elective Paper-6A. Soil, fertilizer and Water analysis**

**OR**

**B. Food Analysis**

**OR**

**C. Analytical method development and validation (2 Credits, 30L T)**

## Major Elective Paper-6- A. Soil, fertilizer and Water analysis (2 Credits, 30L T)

### 1. Analysis of soil [10 L]

a) Sampling of soil, sample preparation, Pre-treatment of Samples and Contamination, Trace Element Analysis, Sub-sampling, Drying Techniques, Milling, Grinding and homogenization, **b) Weighing and Dispensing:** Weighing Errors, Dispensing Errors, **c) Acid-digestion, Ashing and Extraction Procedure:** Acid-digestion and Washing: Acid-digestion of soils, Total soil nitrogen; Microwave acid-digestion, Dry ashing, Nitrate and water-soluble carbohydrate; Extraction Procedures for soils: pH extract ants, Phosphate extract ants, Potassium extractants, Trace element extractants, **d) Analysis of Soil:** Soil Analytical Procedures - Determination of extractable boron, Cation exchange capacity, exchangeable bases and base Saturation, Determination of CEC and exchangeable cations, Measurement of calcium and magnesium by AAS, Measurement of potassium and sodium by flame photometry, Determination of cation exchange capacity (CEC), Determination of effective cation exchange capacity (ECEC), Determination of fulvic and humic acids, Discussion - Determination of available nitrogen, Method-a: Determination of nitrate by selective ion electrode, Discussion - Determination of total mineralized nitrogen, Method-b: Determination of extractable ammonium-N, Method-b: Determination of extractable nitrate-N, Discussion, Determination of organic plus ammonium nitrogen, Method-a: Determination of soil nitrogen by autoanalysis, Method-a: Reduction of nitrate before digestion and colorimetric auto analysis, Method-b: Determination of organic plus ammonium-N by digestion and distillation, Discussion, Determination of soil organic matter, Method-a: Determination of soil organic matter by loss on ignition, Method-b: Determination of easily oxidizable organic C by Tinsley's wet combustion, Discussion 5.8. Determination of pH and lime requirement, Method-a: Measurement of pH, Method-b: Determination of lime requirement, Method-c: Determination of pH in soils with soluble salts, Discussion - Determination of extractable phosphorus, Method-a: Determination of extractable phosphorus (manual method), Method-b: Determination of extractable phosphorus (automated method), Method-c: Determination of resin extractable phosphorus (automated method), Determination of extractable magnesium, potassium and Sodium, Determination of extractable trace elements, Discussion-Determination of extractable sulphur, Method-a. Determination of extractable sulphur (manual method), Method-b. Determination of extractable sulphur (automated method). (**Ref-1:** 17-35, 50-104, **Ref.-2:** 1-14, 71-331)

### 2. Fertilizer Analysis: [6 L]

Discussion -Determination of total nitrogen in presence of nitrate and organic, Method-a: Determination of total nitrogen in presence of nitrate and organic N, with final determination by distillation, Method-b: Determination of total nitrogen in presence Of nitrate and organic N, with final determination by auto-analysis, Discussion - Determination of phosphorus in fertilizers, Method-a. Determination of water-soluble phosphorus(extraction), Method-a: Determination of water-soluble phosphorus, (auto-analysis), Method-a: Determination of water-soluble phosphorus(manual method), Method-b. Determination of 2% citric acid-soluble phosphorus-method for basic slags (Thomas phosphate), Method-c: Determination of total phosphorus in the acid digest from Method-b. with final determination by auto-analysis, Discussion-Determination of potassium in fertilizers, Method-a: Determination of water-soluble potassium, Method-b. Determination of ammonium oxalate-soluble potassium, Method-c: Determination of potassium in the acid digest from, Liming Materials, Determination of the moisture and neutralizing value of liming materials, Determination of fineness of grinding. (**Ref.-1:** 106-123)

### 3. Water Pollution and Measurement of Water Quality [14 L]

a) **Water Pollutants:** Brief explanation of following with respect to their sources and toxic effects -

Inorganic pollutants (Heavy Metals (Cd, Hg, Pb), Metalloids, Organotin Compounds, Inorganic Species (CN<sup>-</sup>, NH<sub>3</sub> and other species), Asbestos), Organic Pollutants (Soaps, Detergents, and Detergent Builders, Pesticides in Water, Polychlorinated Biphenyls), Emerging Water Pollutants, Pharmaceuticals, and Household Wastes, Radionuclides in the Aquatic Environment). (Ref-4: 159-183 supplementary reference-3 and 4)

**b) Analysis: Physical Properties:** Colour (Visible Inspection, Spectrophotometric—Multi-Wavelength Method, Turbidity, Odour, Taste, Acidity, Alkalinity, Calcium Carbonate Saturation, (Introduction, Indices Indicating A Water's Tendency To Precipitate Or Dissolve CaCO<sub>3</sub>, Indices Predicting The Quantity Of CaCO<sub>3</sub> That Can Be Precipitated Or Dissolved), Hardness, Oxidant Demand/Requirement (Chlorine Demand/Requirement, Ozone Demand/Requirement— Batch Method), Conductivity, Salinity. (Ref-3: 2.5, 2.8, 2.12-2.40, 2.48-2.62). **Metal ions:** Introduction, Preliminary Treatment Of Samples (Introduction, Filtration for Dissolved and Suspended Metals, Treatment for Acid-Extractable Metals, Digestion for Metals, Nitric Acid Digestion, Nitric Acid-Hydrochloric Acid Digestion, Nitric Acid-Sulfuric Acid Digestion, Nitric Acid-Perchloric Acid Digestion, Nitric Acid-Perchloric Acid Hydrofluoric Acid Digestion, Dry Ashing, Microwave-Assisted Digestion), Quantitative analysis by AAS, FES and ICPAES: Only general explanation as this part is covered in detail in Analytical spectroscopy Sec-I. (Ref-3: 3.1-3.35, 3.36-3.67, 3.70-3.71, 3.76-3.78, 3.82-3.84, 3.104-3.105). **c) Inorganic non-metal:** Introduction, Determination of Anions By Ion Chromatography, Inorganic Anions By Capillary Ion Electrophoresis; Bromide (phenol red method), cyanide, Chlorine (DPD colorimetric method), Fluoride (ion selective method, complexone method), ammonia (titrimetric method, ions elective method and phenate method), NO<sub>2</sub>- - colorimetric method, NO<sub>3</sub>- (nitrate electrode and Cd reduction method), Organic nitrogen by MicroKjeldahl method, Dissolved oxygen (iodometric and membrane electrode method), phosphate (molybdate – SnCl<sub>2</sub> - colorimetric method), Sulfide (methylene blue and ion selective method), **d) Organic constituents:** Biochemical oxygen demand, Chemical oxygen demand, total organic carbon, phenols (direct photometric method), surfactants. (Ref-1: 4.1-4.14, 4.17, 4.30-4.31, 4.39-4.46, 4.61, 4.72, 4.86-4.90, 4.114-4.120, 4.124 -4.131, 4.139, 4.114, 4.149, 4.156-4.161, 4.181-4.184, 5.5-5.29, 5.49-5.58, supplementary reference-3,4,5 and 6)

#### References:

1. Methods in Agricultural Chemical Analysis: A Practical Handbook, N.T. Faithfull, CABI Publishing, Typeset by Wyvern 21 Ltd, Bristol (2002).
2. Soil Sampling and Methods of Analysis, Edited by M.R. Carter E.G. Gregorich, Canadian Society of Soil Science, Second Edition (2008)
3. Standard methods for the examination of water and waste water, 23rd Ed. Rodger Baird, Andrew Eatson, Eugene Rice, jointly published by: American Public Health Association, American Water Works Association, and Water Environment Federation.
4. Environmental Chemistry, Stanley E. Manahan, Ninth Edition, CRC press, Taylor and Francis, 2010.
5. Handbook of Environmental Analysis Chemical Pollutants in Air, Water, Soil, and Solid Wastes by Pradyot Patnaik, Third Edition, CRC press, Taylor and Francis, 2018.
6. Environmental Chemistry, A. K. Day, New Age Publication Company

#### Learning Objective –

At the end of course students should able to-

1. Define / understand various terms in soil, fertilizer and water analysis.
2. Explain / describe techniques / methods of soil, fertilizer and water analysis.
3. To describe basic principles techniques / methods of soil, fertilizer and water analysis.
4. Explain importance of soil, fertilizer and water analysis.
5. Choose suitable method / techniques to characterize quality of soil, fertilizer and water.
6. Describe / explain results of analysis soil, fertilizer and water.
7. Solve numerical problems on soil, fertilizer and water analysis..
8. Draw conclusion regarding soil, fertilizer and water quality from analytical results.

**OR**

### **Major elective Paper-6- B. Food Analysis (2 Credits, 30L T)**

**1. Introduction to Food Analysis (Ref-1: 1-13) [1 L]**

**2. Sampling and Sample Preparation [1 L]**

Introduction, Selection of Sampling Procedures, Sampling Procedures, Preparation of Samples, Grinding, Enzymatic Inactivation, (Ref-1: 71-80)

**3. Moisture and Total solids Analysis [2 L]**

Introduction, Importance of Moisture Assay, Moisture Content of Foods, Forms of Water in Foods, Sample Collection and Handling, Oven Drying Methods: General Information, Removal of Moisture, Decomposition of Other Food, Constituents, Temperature Control, Types of Pans for Oven Drying Methods, Handling and Preparation of Pans, Control of Surface Crust Formation (Sand Pan Technique), Calculations; Distillation Procedures, Chemical Method: Karl Fischer Titration. (Ref-1 87-96).

**4. Ash Analysis [3 L]**

Introduction: Definitions, Importance of Ash in Food Analysis, Ash Contents in Foods; Methods: Sample Preparation, Plant Materials, Fat and Sugar Products, Dry Ashing, Principles and Instrumentation, Procedures, Special Applications, Wet Ashing, Principle, Materials, and Applications, Procedures, Microwave Ashing, Microwave Wet Ashing, Microwave Dry Ashing, Other Ash Measurements, Comparison of Methods

**5. Analysis of Lipids [6 L]**

a) Definition, Classification, General Considerations, Solvent Extraction Methods: Sample preparation, Solvent selection, Sample Preparation, Solvent Selection, Continuous Solvent Extraction Method: Goldfish Method, Semi contiguous Solvent Extraction Method: Soxhlet Method, Discontinuous Solvent Extraction Methods, Total Fat by GC for Nutrition Labelling (AOAC Method 996.06), Non solvent Wet Extraction Methods, Babcock Method for Milk Fat (AOAC Method 989.04 and 989.10), Gerber Method for Milk Fat, Instrumental Methods, Comparison of Methods. (Ref.-1: 119-130) b) **Characterization of Lipids** (bulk such as oils): Estimation of free fatty acids, Saponification value of oils, iodine value, Determination of acid value of oil, determination of peroxide value of oil, p-anisidine Value and Totox Value, Thiobarbituric Acid Reactive Substances Test, Conjugated Dienes and Trienes, Lipid Oxidation: Evaluating Oxidative Stability, Methods for Lipid Components, Identification and quantification of fatty acids, Problem on quantitative methods. (Ref-1: 241, 246-258, Supplementary-2, 3).

**6. Proteins [6 L]**

a. **Protein Analysis:** Introduction, Classification and General Considerations, Importance of Analysis, Content in Foods, Methods: Following methods with respect to principle, reactions, procedures and applications a) Kjeldahl Method b) Dumas (Nitrogen Combustion) Method, c)

Infrared Spectroscopy, d) Biuret Method e) Lowry Method f) Dye-Binding Methods g) Bicinchoninic Acid Method h) Ultraviolet 280nm, Comparison of Methods. (**Ref-135** – 142, Supplementary-2, 3). **b.**

**Protein Characterization Procedures:** Amino Acid Analysis, Protein Nutritional Quality:

Introduction, Protein digestibility, Protein efficiency ratio, and net protein ratio, Other Protein Nutritional Quality Tests, Assessment of Protein Functional Properties, Determination of net protein utilization, digestibility and biological value, Problem on quantitative methods (**Ref-1:** 271 - 277, Supplementary-2, 3)

#### **7. Carbohydrates: [6 L]**

Introduction, Mono- and Oligosaccharides: Extraction, Total Carbohydrate: Phenol-Sulfuric Acid Method, total reducing sugars by Nelson Somyogi method, Specific Analysis of Mono- and Oligosaccharides - High-performance Liquid, Gas Chromatography, Enzyme Methods, Chromatography, Mass Spectrometry, Thin-layer Chromatography, Polysaccharides: Starch, Total Starch, Degree of Gelatinization of Starch, Degree of Retro gradation of Starch, Non-starch Polysaccharides, Dietary Fibres: Major Components of Dietary Fibre, General Considerations, Methods. (**Ref-1:** 149-169 Supplementary-2, 3).

#### **8. Determination of food preservatives [5 L]**

Definition, SO<sub>2</sub> legislation and determination by Tanners method, Nitrate and nitrites legislation and determination, boric acid legislation and determination, Benzoic acid legislation and determination, 4-hydroxybenzoate legislation and determination, ascorbic acid legislation and determination. Sweeteners: Saccharine identification and determination, Colours: Identification by general methods, Natural colours. Problem on quantitative methods. (**Ref-4:** Relevant pages)

### **References**

1. Food Analysis, Edited by S. Suzanne Nielsen, Fourth Edition, Springer
2. Hand Book of Food Analytical Chemistry: Water, Proteins, Enzymes, Lipids, and Carbohydrates by Edited by Ronald E. Wrolstad, Terry E. Acree, Eric A. Decker, Michael H. Penner, David S. Reid, Steven J. Schwartz, Charles F. Shoemaker, Denise Smith, Peter Sporns, Wiley Interscience, a John Wiley & Sons, Inc., Publication.
3. Biochemical Methods, By S Sadashivan, A. Manickam; Third Edition, New Age International Publishers
4. Pearson's Chemical Analysis of Food

### **Learning Outcomes –**

At the end of course students should able to-

1. Define / understand various terms in food analysis techniques and methods.
2. Explain methods and principles of analysis of i) Food - carbohydrates, proteins, preservatives
3. Select appropriate methods of food analysis for its quality.
4. Select appropriate methods for identification of food from sample.
5. Select and describe the parameters required for food quality.
6. Solve numerical problems on analysis food substances.
7. Interpret food quality from analytical results.
8. Differentiate among the different methods of analysis of substances.

**OR**

### **Major Elective Paper 6C. Analytical method development and validation**

### **1. Assay Validation and Inter Laboratory Transfer [4 L]**

Introduction, fundamental definitions, Essential principles of method transfer, method validation report, the interlaboratory qualification (ILQ) process. (Ref-1;pp 3 to 14)

### **2. Statistical Analysis and analytical Figure of Merit [14 L]**

Introduction, Errors (gross errors, systematic errors, random errors), accuracy, validation parameters: Accuracy, precision, mean and standard deviation, calibration, (linear response functions (linear regression-errors in slope and the intercept, error in the estimate of concentration, standard additions), non-linear response functions and weighted regression analysis, internal standards), selectivity and specificity (chromatographic methods), limits of detections (spectrophotometric methods, chromatographic methods and related techniques, receptor binding assay), limit of quantification, sensitivity, ruggedness and robustness, analyte stability in the sample matrix, how to reduce systematic errors, mean and standard deviation, reliability of results, confidence interval, comparison of results, comparison of two means of two samples, experimental design.(Ref-1:15 to 68, Ref-2, p145-197)

### **3. Overview of World Wide Regulations (4 L), Ref-1: 75 to 98)**

### **4. Specific methods and Applications: Dissolution Studies [4 L]**

Introduction, Dissolution test, Apparatus – USP type –I and II, Sampling and analytical instrumentation, Single point test Vs. Dissolution profile, Calibration, regulatory guidelines, analytical validation, linearity, accuracy, precision, specificity.(Ref-1: 169 to 182)

### **5. Specific Examples [4 L]**

Explain these method w.r.t. method development and validation of specified analyte from the research papers.(Ref-4 to 7)

#### **References**

1. Development and validation of Analytical Methods, Progress Pharmaceutical and Biomedical Analysis, Vol-3, Edited by Chitofer M. Riley and Tomas W. Rosanske (Elvier).
2. Vogel's Textbook of quantitative Chemical Analysis, Sixth Ed., Mendham, Denney, Barnes, Thomas, Pub: Pearson Education.
3. Development and validation of a colorimetric method for the quantitative analysis of thioamide derivatives, R.B. Ali et al., Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 220 (2019) 117154.
4. HPLC Method Development and Validation for Formaldehyde in Enteric Coating of Gelatine Capsules, Journal of Liquid Chromatography, 18(13), 2683-2693 (1995).
5. Development and Validation of Stability Indicating RP-HPLC Method for Analysis Of Lercanidipine In Bulk Drug And Micro emulsion Formulation, Journal of Liquid Chromatography & Related Technologies, 36:143–154, 2013.
6. Development and validation of an LC-MS/MS method for simultaneous quantification of voriconazole and its main metabolite voriconazole N-oxide in human plasma and its clinical application, Journal of Liquid Chromatography & Related Technologies, 40:20, 1047-1053.
7. Development and validation of the spectrophotometric method of butaphosphan determination in veterinary preparations, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 233 (2020) 118171.

#### **Learning Outcomes –**

At the end of course students should able to-

1. Define / understand various terms in method development and validation.
2. Explain instrumentations and methodology in analytical method development and validation
3. Explain / describe basic principles of analytical method development and validation.
4. Explain /Describe applications analytical method development and validation in industry and in analytical laboratory.
5. Apply / select particular method of analysis for sample to be analysed.
6. Solve numerical problems on analytical method development and validation.
7. Develop analytical method for analysis of given sample. Apply statistical treatment to the analytical data. Select appropriate parameters for the development of analytical method.

### Major Elective Paper 7A. Applied Inorganic Chemistry Practical-III

#### Major Elective Paper 7A. Soil, water and fertilizer analysis ( 2 Credits, 60 L , Any 12 Practical's)

Sr. No	Title With Contents (Any 12)
<b>Soil Analysis (Any 4)</b>	
1	Analysis of soil sample for moisture content and organic carbon.
2	Analysis of soil sample for nitrogen by Kjeldahl method.
3	Analysis of soil sample for i) potassium by flame photometry ii) pH and conductance.
4	Analysis of soil sample for phosphorus by colorimetry.(phosphoammonium molybdate method)
5,6	Analysis of soil sample for CEC and ECEC ( $H^+$ , $Al^{3+}$ , $Ca^{2+}$ , $Mg^{2+}$ ) by ion exchange chromatography and flame photometry.
7	Analysis of soil sample for boron by ICP spectrometry.
8	Report writing on soil sampling and digestion process.
<b>Water Analysis (Any 5)</b>	
9	Analysis of waste water sample for i)Total dissolved salt ii) COD
10	Analysis of waste water sample for i) Chloride ii) sulphate
11	Analysis of waste water sample for alkalinity.
12	Analysis of waste water sample for total hardness.
13	Analysis of waste water sample for phosphate by colorimetry.(phosphoammonium molybdate method)
14	Analysis of waste water sample for Pb by gravimetric analysis.
15	Analysis of waste water sample dissolved oxygen by Winkler process.

	<b>Fertilizer Analysis (Any 3)</b>
16	Analysis of fertilizer sample for nitrogen by Kjeldahl method.
17	Analysis of fertilizer sample for potassium by flame photometry and gravimetry.
18	Analysis of fertilizer sample for phosphate by titrimetry.
19	Analysis of fertilizer sample for iron by colorimetry/Spectrophotometry.
20	Data Analysis using Tools like MS Excel, Origin Pro, ChemDraw and MATLAB, Google scholar, chemspider, scifinder, Scopus, reaxys, research gate

## References

1. Methods in Agricultural Chemical Analysis: A Practical Handbook, N.T. Faithfull, CABI Publishing, Typeset by Wyvern 21 Ltd, Bristol (2002).
2. Soil Sampling and Methods of Analysis, Edited by M.R. Carter E.G. Gregorich, Canadian Society of Soil Science, Second Edition (2008)
3. Standard methods for the examination of water and waste water, 23rd Ed. Rodger Baird, Andrew Eatson, Eugene Rice, jointly published by: American Public Health Association, American Water Works Association, and Water Environment Federation.
4. Environmental Chemistry, Stanley E. Manahan, Ninth Edition, CRC press, Taylor and Francis, 2010.
5. Handbook of Environmental Analysis Chemical Pollutants in Air, Water, Soil, and Solid Wastes by Pradyot Patnaik, Third Edition, CRC press, Taylor and Francis, 2018.
6. Environmental Chemistry, A. K. Day, New Age Publication Company

## Learning Outcomes-

At the end of course students should able to-

1. Maintain proper record of analytical data in notebook. Observe personal safety in laboratory and able to handle all chemicals, instruments, etc safely in laboratory.
2. Define / understand various terms involved in practical methods of quantitative analysis.
2. To analyse organic and inorganic materials using appropriate chemical / instrumental methods
3. Explain / describe basic principles of chemical / instrumental methods used for analysis. Able to handle particular instrument according to SOP.
4. Perform analysis of sample with described procedure. Able to handle analytical instruments.
5. Apply / select particular method / instrumental parameters for analysis of given sample.
6. Maintain appropriate reaction conditions as described in procedures.
7. To perform i) selective analysis of particular component from sample. ii) Analysis at trace level from sample.
8. To conclude the results and take the decision regarding quality of sample.
9. To perform calculations and interpret the results.

**OR**

**Major Elective Paper 7B. Food analysis Practical's (2 Credits, 60 L , Any 12 Practica  
Major Elective Paper**

**Major Core Paper-8. Project (4 Credits)**

## **GUIDELINES TO CARRY OUT PROJECTWORK**

1. The main purpose of introduction Project Work at MSc Part II is to make the students familiar with Research Methodology i.e. reference work, experimental work, statistical analysis of experimental data, interpretation of results obtained, writing of project work and compilation of bibliography in proper order. This will not only help train the inquisitive minds of the students, but also inspire them to take up research-oriented higher studies and career.

2. Duration of Project work: -

Development on the nature of the research problem and the infrastructure available in the respective chemistry Departments or Research Institutes or Industries, the duration of Project Work is recommended as follows:-

a. The project work will commence immediately after the conclusion of Semester II of M. Sc Part – I.

b. Each student shall complete a small research project during his/ her academic year of M. Sc Part-II. However, the initial reference work can be started in M.Sc Part- I and summer vacation.

4. Nature of Research Project:-

The following will be considered as the Research Project.

a. Experimental based involving laboratory analytical work, or

b. Industrial training based provided that the candidate has undergone actual hands on training in instrumental analytical techniques.

## **Semester IV**

### **Major Core Paper 1 Advanced Analytical spectroscopic techniques (4 credits 60 L T)**

#### **Section-I: Atomic Spectroscopic Methods [2 credits 30 L T]**

##### **1. Sample preparation techniques [2 L]**

Introduction, aqueous sample, liquid-liquid extraction, Ion exchange, co-precipitation, solid samples: decomposition techniques, microwave digestion, dry ashing, fusion, Extraction procedures: Single extraction, sequential extraction, enzymatic digestion (Ref-1: 17-36, Supplementary reference - 2)

##### **2. Atomic Absorption and emission Spectroscopy [6 L]**

Introduction, Atomic spectra, Instrumentation of AAS: Sample introduction system: Nebulizers, Laser Ablation technique, hydride vapour generators, atomizers: Flame atomizer - premix burner, fuel gases and oxidants, graphite furnace, hydride generator, cold vapour technique, Hollow cathode lamps, spectrophotometers, detectors, Interferences in AAS (spectral and chemical), Quantitative analysis (calibration curve method, standard addition method, internal standard addition method), Practical applications of AAS from Ref-3. (Ref-3: Relevant pages, Supplementary references 4,5)

##### **3. Inductively Coupled Plasma AES and MS [10 L]**

**a. Inductively Coupled Plasma AES:** Introduction to Atomic emission spectroscopy, inductively coupled plasma, Direct current plasma, microwave induced plasma, glow discharge, plasma spectroscopy, spectrometers, Detectors, interferences.

**b. Inductively Coupled Plasma MS:** Fundamental of MS, Inorganic mass spectroscopy, Interface, mass spectrometer, quadrupole mass analyser, detectors, interferences, isotope dilution analysis, mass spectral interpretation. (Ref-1:57-117, supplementary Ref- 6)

**c. Applications:** Forensic analysis of documents, Clinical analysis of blood and urine, (**Ref-1:** Relevant pages). Analysis of metals from waste water sample of ICP-MS method (Ref-2, sec. 3120, 3125)

#### **4. Atomic Fluorescence Spectroscopy [6 L]**

Atomic fluorescence, Apparatus for AFS, EMR source for AFS, LASERS, Cells for AFS, Plasmas-ICP and DCP, Detectors, theory of AFS, Analysis with AFS, Interferences with AFS, Resonant ionization Spectroscopy, LASER enhanced ionization spectroscopy.(Ref-5)

#### **5. Elemental Analysis [2 L]**

Particular analyses, Elemental organic microanalysis, Total nitrogen analysers (TN), Total sulphur analysers, Total carbon analysers, problems on empirical and molecular formula on CHONS analysis. (**Ref. -7:** 441-450)

#### **Reference**

1. Practical Inductively Coupled Plasma spectroscopy, John R. Dean, Wiley India Pvt. Ltd. (AnTs Series book)
2. Standard methods for the examination of water and waste water, 23rd Ed. Jointly published by American Public Health Association, American Water Work Association, Water Environment Federation. 2017.
3. Vogels, Quantitative Chemical Analysis, 6th Ed.
4. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.
5. Introduction to Instrumental Analysis by R. D. Braun
6. Practical Guide to ICP-MS, Edited by Robert Thomas, CRC press, Francis and Taylor.
7. Chemical Analysis Modern Instrumentation Methods and Techniques, Francis Rouessac and Annick Rouessac, Second Edition, John Wiley & Sons Ltd

### **Section-II: Molecular Spectroscopic Methods [2 credits 30 L T]**

#### **1. Molecular Luminescence spectrometry [10 L]**

Introduction, theory of fluorescence and phosphorescence: excited state producing fluorescence and phosphorescence, energy level diagram, rate of absorption and emission, deactivation process, variables affecting fluorescence and phosphorescence, Emission and excitation spectra; Instruments for measuring fluorescence and phosphorescence: Components of Fluorometers and Spectrofluorometers, Instrument Design, Correction and Compensation Schemes, Instrument standardization; Applications of Photoluminescence Methods: Methods for Organic and Biochemical Species, Phosphorometric method, Fluorescence Detection in Liquid Chromatography, Lifetime measurement, Fluorescence imaging; **Chemiluminescence:** The Chemiluminescence phenomenon, measurement of chemiluminescence, analytical applications, problems. (**Ref.-1:**399-426)

#### **2. Electron Paramagnetic Resonance Spectroscopy [12 L]**

**Basic Theory:** general remarks, electron spin and magnetic moment, ESR transitions, Selection rules, g-factor, presentation of spectra, interaction of magnetic dipole with microwave radiations, Larmor precession, resonance phenomenon, relaxation process, transition probability. **Hyperfine Structure:** Nuclear hyperfine splitting, radical containing one proton, spin Hamiltonian, selection rules, radical containing a set of equivalent protons, radical containing a set of multiple protons, radical containing multiple sets of protons ( $I = \frac{1}{2}$ ), radical containing multiple sets of proton ( $I > \frac{1}{2}$ ), Atomic radicals, Origin of hyperfine interaction, sigma radicals, assignments of spectra using Huckel MOs, alternant hydrocarbons, hyperfine splitting constants, second order splitting,

Applications.(Ref-3:Relevant pages, Supplementary Ref-4)

### **3. Electron Spectroscopy for Surface Analysis [8 L]**

Basic principles, x-ray photoelectron spectroscopy, Auger Electron spectroscopy, Instrumentation: ultra-high vacuum, source gun, electron gun, Ion gun, electron energy analysers, Characteristics of Electron spectra: photoelectron spectra, Auger electron spectra, Qualitative and quantitative analysis: qualitative analysis, peak identification, chemical shift, problems with insulating materials, Quantitative analysis: peak and sensitivity factor, composition depth profiling.(Ref-2: 221-250).

#### **References:**

1. Principles of Instrumental Analysis, Skoog, West, Holler, 6th Ed. Cengage Publication.
2. Materials Characterization, introduction to microscopic and spectroscopic techniques, Yang Leng, 2nd Wiley-VCH.
3. Introduction to Magnetic Resonance of Spectroscopy ESR, NMR, NQR, D.N. Sathyanarayana, I. K. International Publishing House Pvt. Ltd.
4. Structural Methods in Molecular Inorganic Chemistry, David W. H. Rankin, Norbert W. Mitzel, Carole A. Morrison, Wiley (John Wiley & Sons, Ltd.), 2013
5. Advanced Instrumental Analysis, Chatwal and anand

#### **Learning Outcomes –**

At the end of course students should able to-

1. Define / understand various terms in atomic absorption, atomic emission, fluorescence, ESR and electron spectroscopy.
2. Explain instrumentation of atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.
3. To describe basic principles of atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.
4. Select appropriate methods for sample treatment in AAS / AES, ICPAES, ICPAES-MS.
5. Explain advantages of ICPAES-MS over AES spectroscopy, fluorescence spectroscopy.
6. Solve numerical problems on analysis all these spectroscopic methods.
7. Interpret ESR spectra, super hyperfine splitting and g value in ESR, and parameters affecting it.
8. Calculate theoretical parameters from ESR data and characterize compound.
9. Solve problems based on atomic absorption, atomic emission, ICPAES, ICPAES-MS, fluorescence, ESR and electron spectroscopy.

## **Major Core Paper 2 Chemical methods of pharmaceutical analysis (4 credits 60 L T)**

### **Section-I: Pharmaceutical Dosage forms and General Methods Analysis [30 L T]**

#### **1. Pharmaceutical Dosage Forms [4 L]**

Capsules: Definition, types of capsules, Tests; Creams: Definition, tests; Ear Drops: Definition, tests; Eye Drops: Definition, tests; Gels: Definition, Inhalation Preparations: Definition, Uniformity of delivered, Number of deliveries per container dose, Uniformity of delivered dose (only); Nasal preparations: Definition and tests; Ointments: Definition and tests; Oral Liquids: Definition, types and tests; Oral Powders: Definition and tests; Parenteral Preparations: Introduction, Injections: Definition and tests, Infusion: Definition and tests; Powder for Injection: Definition and tests; Tablets: Definition, types of tablets and their tests.(Ref-2: 14 - 47), Shelf life of pharmaceutical preparation.

## **2. Chemical Test, Limit Test and Assay [6 L]**

**Important Note:** Write the chemical reaction and explain theoretical basis of the limit tests and assay though it is not given in reference book.

**a) Limit Tests:** Aluminium, Aluminium in Adsorbed Vaccines, Arsenic, Calcium in Adsorbed Vaccines, Chlorides, Heavy metals, Iron, Lead, Potassium, Sulphates, Sulphated Ash, Total Ash, Free Formaldehyde, N-N-Dimethylaniline (Ref-1: 74-80, Ref.- 4, 93-149)

**b) Assays:** Acetyl Value, Acid Value, Cineole, Ester, Ester Value, Hydroxyl Value, Iodine value, Nitrogen, Methoxyl, Nitrite Titration, Peroxide Value, Saponification Value, Assay of Steroids, Unsaponifiable Matter, Assay of Vitamin A, Assay of Vitamin D, Water-(Titration method and azeotropic distillation method), Zinc, Ethanol, Assay of Insulins(Ref-1: 80-99, Ref-3)

## **3. Pharmaceutical Methods of Determination [6L]**

Disintegration Test, Dissolution Test, Uniformity of Weight of Single-Dose Preparations, Uniformity of Content of Single-Dose Preparations, Friability of Uncoated Tablets, Contents of Packaged Dosage Forms, Powder Fineness, Particle Size by Microscopy, Particulate Contamination. (Ref-1: 175-188)

## **4. Microbiological Assay of Pharmaceuticals [8 L]**

Biological assay in general, **a) Agar diffusion assay – Quantitative basis:** Introduction, The theory of zone formation, what happens in practice, principles of calculation of potency estimate; **b) The Theory and Practice of Tube Assay- Growth promoting substances:** Introduction, the mode of action of growth limited by amino acids, growth limited by vitamins, production of acid by lactobacilli, clinical factor in the assay of growth promoting substances; **c) The Theory and Practice of Tube Assay-Growth Inhibiting Substances:** Introduction, measurement of response, the forms of response line, historical development of the turbidimetric method, linearization of sigmoid curve, the quantitative theory of microbial growth and inhibition, a practical determined log dose – response curve, factor affecting final cell count, the influence of temperature, the influence of time, **d) What do we want assay:** pharmacopeial intension and control of antibiotic bulk materials, control in routine in manufacture, Research and development; **d) General Practical Aspects of Microbiological Assay:** Introduction, test solutions (weighing – sample of unknown, dilution of primary solution to test level, problem with very dilute solutions, the assay medium), selection of Latin squares and plating routine, Aspects of technique (the test organism, inoculating the medium, assay plate, assay tube, diluents, the sample, test solution and the effect of contamination, application of test solution-agar diffusion assay, application of test solution-turbidimetric assay; Calculation of potency, **e) Standard and reference materials**( Ref-4: 1, 9-18, 23-35, 37-56, 59-64, 65-77, 79-84, Ref-1: 45-52)

## **Section-II: Analysis of Raw Materials and Active Ingredients [30 L T]**

### **1. Introduction to Pharmaceutical Analytical Chemistry [2 L]**

Introduction, Official European Pharmacopoeia definitions, Pharmaceutical Analytical Chemistry, Manufacture of Pharmaceuticals, Development of New Drugs, Use of Pharmaceuticals (**Ref-3: 1-7**)

### **2. Marketing Authorizations, Pharmaceutical Manufacturing, and International Pharmacopoeias [2 L]**

Introduction, Marketing Authorization and Industrial Production, Pharmacopoeias, Life Time of Pharmaceutical Preparations and Ingredients. (**Ref.3: 9-14**)

### **3. Chemical Analysis of Pharmaceutical Ingredients [14 L]**

Pharmaceutical Ingredients, Production, and Control, Pharmacopoeia Monographs, Melting point capillary method, (monograph on paracetamol and acepromazine malate tablet, acetaminophen,

acetaminophen capsules, castor oil virgin, cefaclor), Impurities in Pharmaceutical Ingredients: Impurities in Pure Chemical Ingredients, Impurities in Organic Multi-Chemical Ingredients; Identification of Pharmaceutical Ingredients: IR Spectrophotometry (identification of ibuprofen, Identification of spironolactone) , UV-Vis Spectrophotometry (Identification of mianserin hydrochloride), Thin-Layer Chromatography (Identification of metrifonate), Melting Point, Optical Rotation (Optical rotation for simvastatin), Liquid Chromatography (Identification of calcitriol), Chloride (Identification of chloride in chlorcyclizine hydrochloride) and Sulfate, Identification, Impurity Testing of Pharmaceutical Ingredients (Pure Chemical Ingredients): Appearance of Solution (Appearance of solution for ibuprofen), Absorbance (Absorbance and color of solution of esomeprazole magnesium) pH and Acidity or Alkalinity (pH of esmolol hydrochloride, Acidity or alkalinity of dopamine hydrochloride), Related Substances (Related substances according to Ph. Eur. for omeprazole), Residual Solvents (Limit of acetone in olmesartan medoxomil), Foreign Anions (Test for foreign chlorides and sulphates in furosemide), Sulphated Ash (Residue on ignition for acetaminophen), Elemental Impurities (Test for foreign zinc in human insulin), Loss on Drying (Loss on drying for paracetamol), Water (Determination of water in ephedrine), Identification and Impurity Testing of Organic Multi-Chemical Ingredients: Oxidizing Substances. **Only importance of the should be explained** - Acid Value, Hydroxyl Value, Iodine Value, Peroxide Value, Saponification Value, Unsaponifiable Matter), Other Tests (Chromatographic profile for peppermint oil), Assay of Pharmaceutical Ingredients, Aqueous Acid–Base Titration (Assay of omeprazole, amitriptyline hydrochloride, ephedrine hydrochloride, ephedrine), Non-Aqueous Acid–Base Titration (metronidazole benzoate, lidocaine), Redox Titrations (ferrous fumarate), Liquid Chromatography (Assay of simvastatin), UV-Vis Spectrophotometry (Assay of hydrocortisone). (**Ref-3:** 305-388)

#### **4. Chemical Analysis of Pharmaceutical Preparations [12 L]**

Chemical Analysis of Pharmaceutical Preparations, Monographs and Chemical Analysis (BP monograph for paracetamol tablets), Identification of the API: Identification by IR Spectrophotometry (Identification of aspirin, fluoxetine in fluoxetine hydrochloride oral solution, Identification of mupirocin in mupirocin calcium nasal ointment), Identification by Liquid Chromatography (Identification of fluoxetine in fluoxetine hydrochloride, droperidol in droperidol injection, Beclomethasone Dipropionate in Beclomethasone Dipropionate Ointment), Identification by UV-Vis Spectrophotometry (Identification of Diazepam in Diazepam Tablets, Flupentixol Decanoate in Flupentixol Decanoate Injection, Miconazole in Miconazole Nitrate Cream), Assay of the Active Pharmaceutical Ingredient, Assays Based on Liquid Chromatography (Assay of Omeprazole, Fentanyl in Fentanyl Citrate Injection, Assay of Hydrocortisone in Hydrocortisone Ointment), Assays Based on UV Spectrophotometry (Assay of Paracetamol in Paracetamol Tablets, Assay of Doxapram in Doxapram Hydrochloride Injection), Assays Based on Titration (Assay of Fe<sup>2+</sup> in Ferrous Fumarate Tablets, Diphenhydramine in Diphenhydramine Hydrochloride Oral Solution), Chemical Tests for Pharmaceutical Preparations, Test for Related Substances (Related Substances in Paracetamol Tablets), Uniformity of Content (Uniformity of Content for Phenindione Tablets), Dissolution. (**Ref-3:** 391-332)

#### **References**

1. Indian Pharmacopeia Volume I, 7th Ed
2. Indian Pharmacopeia Volume II, 7th Ed
3. Introduction to Pharmaceutical Analytical Chemistry, Stig Pedersen-Bjergaard, Bente Gammelgaard, Trine Grønhaug Halvorsen, Second Edition, Wiley (2012).

4. Pharmaceutical Chemical Analysis: Methods for Identification and Limit Tests, Ole Pedersen, CRC press. Taylor & Francis Group, 2006.

### Learning Objective –

At the end of course students should be able to-

1. Define / understand various terms in pharmaceutical raw material and finished product analysis.
2. Explain various pharmaceutical dosage forms and types of raw materials used.
3. To describe basic principles of methods of pharmaceutical analysis according to IP.
4. Explain importance of particular test in pharmaceutical raw material and finished product analysis.
5. Perform and explain importance of limit tests, identification tests and microbiological limit test of raw materials and finished products.
6. Solve numerical problems on analysis of pharmaceutical raw material and finished product analysis.
7. Interpret IR spectra, HPLC chromatogram, UV-Visible spectra of pharmaceutical materials.
8. To perform total analysis of pharmaceutical raw material and finished product analysis according to IP / BP / USP.
9. Standardize analytical instruments according to IP / BP / USP.
10. Take a decision on the basis of analytical results regarding the quality of raw materials so that material can be accepted for production or rejected.

### Major Core Paper-3-Applied Physical Chemistry Practical-IV

Applied Physical Chemistry Practical IV (2 Credits, 60 L Any 12 Practical)	
Unit No.	Title with Content
1.	<b>Spectrophotometry / Colorimetry Technique</b> 1. Determination of stability constant of ferric thiocyanate complex by Ostwald method using spectrophotometer.
2.	<b>Conductometry Technique</b> 1. Determination of boric acid by Conductometry. 2. Determination of acid and salt from mixture of weak and strong acid and salt.
3.	<b>Potentiometry Technique</b> 1. Determination of commercial vinegar by potentiometric titration.
4.	<b>Flame photometry Technique</b> 1. Determination of Na/K from water sample by internal standard method using flame photometry 2. Determination of calcium from dairy whitener by flame photometry.
5.	<b>Photofluometry Technique</b> 1. Analysis of Riboflavin from vitamin supplementary capsules / syrup / tablet sample by standard addition method using Photofluometry. 2. Estimation of quinine sulphate from tablet by calibration curve and its confirmation by standard addition method.
6.	<b>Polarimetry Technique</b> 1. Determination of purity of sugar sample by optical rotation method using polarimetry.

7.	<p style="text-align: center;"><b>Turbidimetry Technique</b></p> <p>1. Estimation of <math>\text{SO}_4^{2-}</math> from water or saline sample or food sample by calibration curve method using nephelometry.</p>
8.	<p><b>Atomic Absorption Spectroscopy Technique</b></p> <p>1. Estimation of micronutrient from food by AAS (any two elements such as Fe, Cu, Zn, Mo, B, Mn).</p>
9.	<p><b>Polarography Technique</b></p> <p>1. Determination of Cu and Zn in brass alloy by polarography.</p>
10.	<p><b>Thermogravimetry Analysis Technique</b></p> <p>1. Estimation of Ca and Mg from form the mixture their oxalate by recording their TGA curve.</p>
11.	<p><b>Gas Chromatography Technique</b></p> <p>1. Analysis of Alcohol from wine by GC</p> <p><b>References:</b></p> <ol style="list-style-type: none"> <li>1. Environmental Chemistry by A.K. De 3rd Ed. Wiley Eastern Ltd. 1) Introduction to Instrumental Analysis by R. D. Broun, Mc Graw Hill (1987).</li> <li>2. Instrumental methods of chemical analysis by H. Willard, L. Merritt, J.A. Dean and F.A. Settle. Sixth edition CBS (1986).</li> <li>3. Fundamentals of Analytical Chemistry, 6th edition, D.A. Skoog, D.M. West and F.J. Holler, Saunders college publishing.</li> <li>4. Principles of Instrumental Analysis, Skoog, Holler, Nieman, (Sixth Ed.)</li> <li>5. Vogel's Textbook of Quantitative analysis 6th Ed.</li> <li>6. Modern analytical techniques in the pharmaceutical and bio analysis <b>By</b> Dr. Istvan Bak (Book Available Online).</li> <li>7. Preparative chromatography Chrome Ed. book series, Raymond P. W. Scott (free e book available on internet).</li> <li>8. Extraction technique in analytical science, John R. Dean, Wiley (2009).</li> <li>9. Practical HPLC method Development, Snyder, Kirkiand, Glajch, Wiley India Pvt. Ltd</li> </ol>

Sr. No.	Aims and Objectives
	Student should understand and learn -
1	Preparation of various low concentration solutions in ppm level.
2	Handling of instrument with taking all operation knowledge.
3	Standardization and Operations of different instruments.
4	Analysis of different commercial compounds using different instrumental techniques.

## Learning Outcomes:

Sr. No.	Learning Outcomes
After studying this course -	
1	Prepare solutions of various concentrations (like molar, normal, ppm, percentage etc.) solution.
2	Calibrate and use colorimeter, uv-visible spectrophotometer, photofluorometer, nephelometer, flame photometer etc.
3	Perform experiments using above instruments.
4	Analyse different commercial compounds.

## Major Core Paper-4-Applied Organic Chemistry Practical-IV

Applied Organic Chemistry Practicals-IV ( 2 Credits, 60 L , Any 12 Practicals)		
Unit No.	Title with Content	No. of Practicals
<b>I</b>	<b>Table Work (Compulsory Practical)</b> 1. Theoretical basic of method development and validation: Accuracy, Precision, Noise level, Detection limit, Quantitation limit, Calibration curve and standard addition method 2. Expression of results: Calculations of mean, standard deviation, error and absolute and relative errors 3. Regression analysis of calibration curve and its importance	<b>1</b>
<b>II</b>	<b>Preparation of Derivatives for Aldehyde/Ketone (Gravimetry)</b> 3. Preparation of 2,4-DNP Derivative ( <b>Any one compound</b> ) 4. Preparation of Oxime Derivative ( <b>Any one compound</b> ) 5. Preparation of Semi carbazone Derivative ( <b>Any one compound</b> )	<b>3</b>
<b>III</b>	<b>Spectrophotometer (Any three Practicals)</b> 1. Estimation of Aspirin 2. Estimation of Lycopene from Tomato/Carrot/Watermelon or any other source of lycopene 3. Estimation of total carbohydrate by anthrone method 4. Estimation of Cholesterol by Ferric Chloride method 5. Estimation of Salicylic acid 6. Estimation of Glucose	<b>3</b>
<b>IV</b>	<b>Isolation of Natural Products (Any four Practicals)</b> 3. Isolation of Cinnamaldehyde from Cinnamon 4. Extraction of Piperine from <i>Piper nigrum</i> (Black Pepper) 5. Extraction of Trimyristin from <i>Myristica fragrance</i> (Nutmeg) 6. Estimation of amino acids by Paper/ Thin Layer Chromatography 7. Extraction of natural essential oils by solvent extraction / Steam Distillation 8. Extraction of alkaloids from green tea	<b>4</b>
<b>V</b>	<b>Nanomaterial</b>	<b>1</b>

	Preparation and analysis of any one nanoparticle (X-Ray Diffraction, SEM and TEM information of nanoparticles should be studied.)	
	<b>References:</b> 1. Vogel Textbook of Quantitative Chemical Analysis, 6 <sup>th</sup> Edition 2. Lab. Manual: Manual of methods of analysis of Foods, Vegetables- Fruit and vegetable products 3. Indian Pharmacopeia Volume I, 7 <sup>th</sup> Edition 4. Indian Pharmacopeia Volume II, 7 <sup>th</sup> Edition 5. Indian Pharmacopeia Volume III, 7 <sup>th</sup> Edition 6. Post Graduate Chemistry Practicals–S.S.Kelkar, H.N.Patel, S.P.Turakhiya,A.G.Gadre, Himalaya Publishing House. 7. Any other relevant reference can be included	

#### Aims and Objectives of the Course-

Sr. No.	Aims and Objectives
Student should understand and learn -	
1	Basic methods development and validation and analysis of calibration curve
2	Preparation of derivatives of aldehyde and ketone
3	Spectroscopic estimations of drugs and some natural products
4	Isolation of some natural products by steam distillation and solvent extraction method

#### Learning Outcomes:

Sr. No.	Learning Outcomes
After studying this course -	
1	Student will be able to understand basic methods of development and validation
2	Student will be able to understand preparation of derivatives of aldehyde and ketones
3	Student will be able to estimate some natural products and drug molecules
4	Student will be able to estimate some natural products in laboratory by solvent extraction

**Major elective -5A. Analytical techniques of polymer, paints and pigments**

OR

**5B. Laboratory Automation and Sensor Based Techniques**

OR

**5C. Forensic analytical chemistry (2 Credits, 30 L T)**

## **Major elective -5-A. Analytical techniques of polymer, paints and pigments (2 Credits, 30 L T)**

### **1.Introduction to polymers: (2 L)**

Brief history to polymers, how polymers are made, classification of polymers.

### **2. Analysis and testing of polymers: (12 L)**

- a) Chemical analysis of polymers: X-ray diffraction analysis, thermal analysis, TGA, DTA.
- b) Physical testing of polymers: Mechanical properties, Fatigue testing, impact testing, tear resistance, hardness, abrasion resistance.
- c) Thermal properties: Softening temperature, flammability.
- d) Optical properties: transmittance, colour, gloss, haze and transparency.
- e) Electrical properties: dielectric constant and loss factor, resistivity, dielectric strength, electronic properties.
- f) Chemical properties: resistance to solvents, vapour permeability, weathering.

### **3. Measurement of molecular weight and size: (6 L)**

End group analysis, colligative properties measurements, solution viscosity and molecular size.

### **4. Analysis of Paints and Pigment (10-L, Ref-1)**

Introduction, test on the total coating, water content, separation of pigment binder, and thinner of solvent type coating, separation of pigment binder, and thinner of latex paints, Identification of the binder, Identification of polymer resins and oils, Identification of plasticizer, Analysis of the vehicle, Identification and Analysis of pigments, Identification of inorganic pigments, Analysis of white and tinted pigments outline of general procedure, HCL insoluble, Titanium dioxide, total lead, acid soluble Al and Fe, acid soluble calcium, total zinc, antimony oxide, total sulfate, total carbonate) analysis of colored pigments, Black pigments, other pigments, identification and analysis of thinners.

### **References**

- 1) Textbook of polymer science 3rd edition by F.W.Billmeyer (1994).
- 2) Principles of polymer systems by F. Rodrigue, Tata Mc Graw Hill, New Delhi.
- 3) Principles of polymer systems by P.J.Flory, Cornell University press, New York.
- 4) Polymer chemistry-an introduction Seymour-Carraher-Marcel Dekker. Inc.New York.
- 5) Polymer Science by V.R. Gowarikar, N.B. Vishvanathane, New Age International Ltd. Publisher (1998)
- 6) Polymer Science by Vasant Gowarikar, Wiley Eastern New York (1998).
- 7) Principle of polymer science, Bahadur and sastri, Narosa publishing house.

## **Major elective -5B. Laboratory Automation and Sensor Based Techniques (2 Credits, 30 L T)**

### **1. Automated Analysis: (08 L, Ref. 3.)**

Automated laboratory analyses, Laboratory apparatus- Continuous flow analysers, Flow injection analysers, Discrete-sample analysers, Centrifugal force analysers, Automatic titrations, Process control, process control analysers.

### **2. Miniaturized Analytical systems (04 L, Ref. 4.)**

Introduction, Concept, theory of miniaturization, microfabrication, silicon and glass micro matching, polymer replication technology, miniaturized analytical components, sampling and sample pre-treatment, system integration, serial integration, parallel integration, commercialization.

### **3. Chemical Sensors (06 L, Ref. 4)**

Introduction, definitions, Classification of chemical sensors, descriptions of chemical sensors (electrochemical sensors, potentiometric sensors, voltammetric chemical sensors, sensors based on conducting properties), Optical sensors (light guides, the evanescent wave, design of fiber optic sensor, indicator mediated sensor), Calorimetric sensors (catalytic gas sensor, thermal conductivity sensor), mass sensor (piezoelectric quartz crystal resonator, surface acoustic wave sensor).

#### **4. Biosensors in analysis (06 L, Ref. 4)**

Introduction, producing biological surface, Achievement of bio transduction (Amperometric, potentiometric, optical).

##### **References:**

- 1) Standard methods of chemical analysis, Sixth Edition, F.J. Welcher.
- 2) Quantitative Inorganic Analysis including Elementary Instrumental analysis, By A. I. Vogel, 3ed, ELBS, 1964.
- 3) Instrumental methods of analysis, R. D. Braun
- 4) Analytical Chemistry, Ed. by Kellner, Mermet, otto, Valcarcel, Widmer, Second Ed. Wiley –VCH

##### **Learning Outcomes –**

At the end of course students should able to-

1. Define / understand various terms in – i) Laboratory automation and sensors, chemical and biosensors
2. Explain instrumentation of automated laboratory analysis and sensors.
3. To describe basic principles of automated laboratory analysis and sensors.
4. Explain importance of automated laboratory analysis and sensors.

### **Major elective -5C. Forensic analytical chemistry (2 Credits, 30 L T)**

#### **1. General Drugs, Other Chemicals [9L]**

Introduction, Pharma drugs [barbiturates, benzodiazepine & other pharma drugs],

**Drug abuse** in sports & Date rape drugs: Introduction, common prohibited substances, analytical approach, Forensic Pharmacological studies, Ingestion of drugs, absorption, distribution, metabolism, pathways of drug metabolism, drug metabolism and drug toxicity, excretion of drugs, detection of drugs on the basis of their Metabolic studies.

**Solvent Abuse** [chlorinated hydrocarbons, Aromatic hydrocarbons, alcohols, glycols, fuel and fuel additives]: absorption, distribution, and metabolism, psychological & clinical effects.

**Analysis:** collection of samples, distillation & extraction, Analysis by GC, HPLC.

#### **2. Narcotic Drugs and Psychotropic Substances [9L]**

Introduction to narcotic drugs, Analysis of Narcotic Drugs and Psychotropic Substances, Classification of Narcotics and other drugs, Analytical techniques for identification of drugs. Characterization and synthesis of 1) Narcotics- heroin and cocaine. 2) Stimulants- caffeine, amphetamines. 3) Depressants- Barbiturates, Benzodiazepines analysis of NDPS evidence by various procedures prescribed by U.N. Manual, DFS manual, spot tests, microcrystal tests, extraction methods, TLC, UV-Vis spectrophotometry, IR spectrophotometry, GC-HPLC, MS, GC-MS, NMR and XRD as exemplified by cocaine, cannabis, amphetamines, opiates and hallucinogens (LSD, psilocybine and mescaline), evidence handling & sampling techniques, clandestine laboratory investigation and designer drugs.

#### **3. Fingerprinting & Other Impressions [12L]**

Fingerprint: Nature, Location, Classification, Types, Patterns of Fingerprints, Poroscopy & Edgeoscopy, Classification of Fingerprints: Henry's Classification, Single Digit Classification, Extended Henry's System, Types of Fingerprints [Latent, Patent and Plastic], Invisible Fingermarks

development methods [Powder methods, Fuming methods, Chemical Methods, etc.] Recent techniques [Digital Imaging & Enhancement, Laser & other radiation-based techniques, Preservation and photography of fingerprints on various surfaces. Ridge counting, Ridge tracing, Minutiae Identification & Matching [Manual and Automated: AFIS]. Palm Prints: Nature, Location, Types, Classification, Development, Lifting, Evaluation, Analysis, Forensic Significance. Footprints: Importance, Gait pattern analysis, Evaluation and analysis of various casts. Electrostatic lifting of latent footprints and comparison with reference sample. Tyre marks / prints and skid marks and comparison with control samples. Cheiloscropy: Nature, location, collection and evaluation of lip print. Ear prints: Introduction, growth & development, evaluation and analysis of ear print.

### References:

1. <https://epgp.inflibnet.ac.in/Home/ViewSubject?catid=16> Paper 03: Fingerprint and other impression
2. <https://epgp.inflibnet.ac.in/Home/ViewSubject?catid=16> Paper 09: Drug of Abuse
3. Krishnamurthy, R., Introduction to Forensic Science in Crime Investigation, 2011, Selective & Scientific Books, New Delhi.
4. Clark, E.G.C.; Isolation and Identification of Drugs, Vol. I and Vol. II, Academic Press, (1986).
5. Moenssens: Finger Prints Techniques, 1975, Chitton Book Co., Philadelphia, New York.
6. Mehta, M. K. : Identification of Thumb Impression & Cross Examination of Finger Prints, 1980 N. M. Tripathi (P) Ltd. Bombay.
7. Cummins & Midlo : Finger Prints, Palms and Soles, 1943, The Blakiston office London
8. Sharma B. R. : Footprints, Tracks and Trials. 1980. Central Law Agency. Allahabad
9. Iannarelli, A V; Ear Identification, Forensic Identification series, Paramount (1989)
10. Saxena's : Saxena's Law & Techniques Relating to Finger Prints, Foot Prints & Detection of Forgery, Central Law Agency, Allahabd (Ed. A.K. Singla).
11. Menzel, E Roland; Fingerprint detection with lasers, Marcel Dekker, NY (1999)

### Learning Outcomes:

1. Identification of Type of Drug.
2. Expertise in handling UV, IR, GC and HPLC.
3. Interpretation of data and comparative study with literature.
4. Crime investigation of drug abuse.
5. Methods of development of fingerprint.
6. Role of Fingerprinting in investigation.

### Major elective -6A. Applied Inorganic Chemistry Practical-IV

OR

### 6B. Applied analytical chemistry practical (2 Credits, 60 L)

#### Major Elective Paper 6A. Applied Inorganic Chemistry Practical-IV ( 2 Credits, 60 L , Any 12 Practical's)

Sr. No	Title With Contents (Any 12)
1	Determination Of Aluminium And Magnesium From Magnalium Alloy.
2	Analysis Of Bronze With Respect To Copper And Tin.
3	Analysis Of Dolomite Ore For Ca, Mg And Silicate Material.
4	Determination Of Nickel And Chromium From Nichrome Alloy.
5	Analysis Of Bauxite Ore for Si and Al.

6	Analysis Of syndet for Fe content.
7	Estimation of Total Casein from milk.
8	Reporting, Documentation And Presentation- Scientific Document; Organization and writing of research papers, short communications, review articles, monographs, peer reviewing, ethics in publishing, predatory journals and publishers, technical and survey reports, authored book and edited books and dissertation.
9	Estimation of iodine value of edible oil.
10	Estimation of Total lactose from milk.
11	Adulteration Tests For Milk And Milk Products.
12	Scientific Software's In Research Design - Using advanced search techniques, web resources, e-journals, e-books, journal access, subscribing TOC alerts, hot articles, citation index, h-index and i-index, Impact factor.
13	Estimation of Ca from plaster of Paris.
14	Estimation of Mg from talcum powder.
15	Analysis of copper ferrite ( $\text{CuFe}_2\text{O}_4$ ) and determine amount of Copper and iron volumetrically.
16	Determination of zinc and chromium from pigment/raw material
17	Estimation Of Chlorophyll Pigment From Leaves.
18	Estimation Of Available Chlorine From Bleaching Powder.
19	Determination of Titanium from pigment/raw material.
20	Industrial visit and report writing.

### References:

1. Quantitative Inorganic Analysis including Elementary Instrumental Analysis by A. I. Vogel, 3<sup>rd</sup> Ed. ELBS (1964)
2. Standard methods of chemical analysis by F. J. Welcher
3. Environmental Chemistry by A. K. De
4. Biochemical Methods, Sadashivam and Manickem, Narosapublication
5. Indian Pharmacopoeia volume –I and II
6. Experiments in chemistry by D. V. Jahagirdar, Himalaya publication
7. Practical Pharmaceutical Chemistry, 4<sup>th</sup> Ed. part-2, Beckett, Stenlake
8. Standard Instrumental methods of Chemical Analysis, F. J. Welcher

### Learning Outcomes-

At the end of course students should able to-

1. Maintain proper record of analytical data in notebook. Observe personal safety in laboratory and able handle all chemicals, instruments, etc. safely in laboratory.
2. Define / understand various terms involved practical methods of quantitative analysis.
2. To analyse organic and inorganic materials using appropriate chemical / instrumental methods
3. Explain / describe basic principles of chemical / instrumental methods used for analysis. Able to handle particular instrument according to SOP.
4. Perform analysis of sample with described procedure. Able to handle analytical instruments.

5. Apply / select particular method / instrumental parameters for analysis of given sample.
6. Maintain appropriate reaction conditions as described in procedures.
7. To perform i) selective analysis of particular component from sample. ii) Analysis at trace level from sample.
8. To conclude the results able to take the decision regarding quality of sample.

**OR**

**6B. Applied analytical chemistry practical (4 Credits, 60 L)**

**Major Core Paper-7. Project (6 Credits)**

**GUIDELINES TO CARRY OUT PROJECTWORK**

1. The main purpose of introduction Project Work at MSc Part II is to make the students familiar with Research Methodology i.e. reference work, experimental work, statistical analysis of experimental data, interpretation of results obtained, writing of project work and compilation of bibliography in proper order. This will not only help train the inquisitive minds of the students, but also inspire them to take up research- oriented higher studies and career.

2. Duration of Project work: -

Development on the nature of the research problem and the infrastructure available in the respective chemistry Departments or Research Institutes or Industries, the duration of Project Work is recommended as follows:-

a. The project work will commence immediately after the conclusion of Semester II of M. Sc Part – II.

b. Each student shall complete a small research project during his/ her academic year of M. Sc Part- II. However, the initial reference work can be started in M.Sc Part- II and summer vacation.

4. Nature of Research Project:-

The following will be considered as the Research Project.

a. Experimental based involving laboratory analytical work, or

b. Industrial training based provided that the candidate has undergone actual hands on training in instrumental analytical techniques.