

#### 8 SEM PRACTICE QUESTIONS

ADVANCED INSTRUMENTATION TECHNIQUES

#### UNIT-I

#### Nuclear Magnetic Resonance spectroscopy

Principles of H-NMR and C-NMR, chemical shift, factors affecting chemical shift, coupling constant, Spin - spin coupling, relaxation, instrumentation and applications

**Mass Spectrometry**- Principles, Fragmentation, Ionization techniques – Electron impact, chemical ionization, MALDI, FAB, Analyzers-Time of flight and Quadrupole, instrumentation, applications

- 1. Define chemical shift in NMR spectroscopy.
- 2. What is the unit of measurement for chemical shift?
- 3. Name any two ionization techniques used in mass spectrometry.
- 4. What does MALDI stand for?
- 5. State the principle of proton NMR spectroscopy.
- 6. What is spin-spin coupling?

- 1. Explain the principle of <sup>1</sup>H-NMR spectroscopy.
- 2. Write a short note on the factors affecting chemical shift.
- 3.Describe the working of a quadrupole mass analyzer.
- 4. Compare electron impact and chemical ionization techniques.
- 5.Discuss the concept of coupling constant in NMR and its significance.
- 6.Explain the principle and application of Time-of-Flight (TOF) analyzer.

- Discuss the principles of proton (<sup>1</sup>H) and carbon (<sup>1</sup><sup>3</sup>C) NMR spectroscopy. Include chemical shift, spin-spin coupling, and relaxation in your answer.
- 2. Describe in detail the instrumentation and applications of NMR spectroscopy.
- 3. Explain the principle of mass spectrometry. Discuss various ionization techniques such as Electron Impact, Chemical Ionization, MALDI, and FAB.
- 4. Compare and contrast the Time-of-Flight and Quadrupole mass analyzers with suitable diagrams and applications.
- 5. Write in detail about the fragmentation patterns in mass spectrometry and their role in structure elucidation.

#### UNIT-II

#### **10 Hours**

**Thermal Methods of Analysis**: Principles, instrumentation and applications of ThermogravimetricAnalysis (TGA), Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC)

X-Ray Diffraction Methods: Origin of X-rays, basic aspects of crystals, X-

ray

Crystallography, rotating crystal technique, single crystal diffraction, powder diffraction, structural elucidation and applications.

- 1. What is the principle of Thermogravimetric Analysis (TGA)?
- 2. Name one key difference between DTA and DSC.
- 3. What does DSC measure in a sample?
- 4. Define X-ray crystallography.
- 5. What is the source of X-rays in XRD instruments?
- 6. Mention any one application of X-ray diffraction methods.

- 1.Compare Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA).
- 2.Explain the principle and instrumentation of Differential Scanning Calorimetry (DSC).
- 3.Write a short note on the applications of thermal analysis in pharmaceuticals.
- 4. Describe the principle and process of powder X-ray diffraction.
- 5.Explain the importance of single crystal diffraction in structural elucidation.
- 6.Outline the rotating crystal technique used in X-ray diffraction studies.

- 1. Describe in detail the principles, instrumentation, and applications of TGA, DTA, and DSC. Compare them with examples.
- 2. Discuss the role of thermal methods of analysis in characterizing pharmaceutical substances.
- 3. Explain the origin of X-rays and discuss the basic aspects of crystals relevant to X-ray crystallography.
- 4. Describe various X-ray diffraction methods: rotating crystal, single crystal diffraction, and powder diffraction. Include instrumentation and applications.
- 5. Explain how X-ray diffraction is used in the structural elucidation of crystalline materials.
- 6. Analyze the role of DSC and TGA in determining the purity and thermal stability of drug substances.

#### UNIT-III Calibration and validation-as per ICH and USFDA guidelines Calibration of following Instruments

Electronic balance, UV-Visible spectrophotometer, IR spectrophotometer,

Fluorimeter, Flame Photometer, HPLC and GC

- 1. What is the purpose of instrument calibration?
- 2.Expand ICH and USFDA.
- 3.Name any two instruments that require calibration in a pharmaceutical lab.
- 4. What is validation according to ICH guidelines?
- 5. Mention one parameter checked during HPLC calibration.
- 6.Define the term "traceability" in the context of calibration.

- 1.Explain the difference between calibration and validation with examples.
- 2.Briefly describe the ICH guidelines for equipment calibration.
- 3.Write a short note on the calibration of a UV-Visible spectrophotometer.
- 4.Describe the steps involved in calibrating an electronic balance.
- 5.What are the key parameters assessed during calibration of an IR spectrophotometer?
- 6.Outline the importance of fluorimeter and flame photometer calibration in pharmaceutical analysis.

- 1. Discuss the concepts of calibration and validation as per ICH and USFDA guidelines. Why are they important in pharmaceutical analysis?
- 2. Describe in detail the calibration procedure for HPLC and GC systems.
- 3. Explain the step-by-step calibration method for any four instruments from the following: electronic balance, UV-Vis spectrophotometer, IR spectrophotometer, fluorimeter, flame photometer.
- 4. Analyze the regulatory significance of calibration and validation in maintaining analytical instrument performance.
- 5. Write detailed notes on the qualification of analytical instruments (DQ, IQ, OQ, PQ) with reference to ICH guidelines.

#### UNIT-IV

Radio immune assay: Importance, various components, Principle, different methods, Limitation and Applications of Radio immuno assay Extraction techniques: General principle and procedure involved in the solid phase extraction and liquid-liquid extraction

- 1. What is the basic principle of Radioimmunoassay (RIA)?
- 2. Name any one application of RIA.
- 3. List any two components of a typical radioimmunoassay system.
- 4. Define solid-phase extraction (SPE).
- 5. What is the main purpose of liquid-liquid extraction?
- 6. Mention one limitation of RIA.

- 1.Describe the principle and importance of Radioimmunoassay in pharmaceutical analysis.
- 2. Write a short note on the components involved in Radioimmunoassay.
- 3. Explain the general procedure of solid-phase extraction (SPE).
- 4.Differentiate between solid-phase extraction and liquid-liquid extraction.
- 5. What are the limitations of using RIA in analytical procedures?
- 6.Briefly explain the principle and applications of liquid-liquid extraction.

- 1.Explain in detail the principle, components, types, limitations, and applications of Radioimmunoassay (RIA).
- 2.Discuss the importance of RIA in clinical and pharmaceutical analysis. Include a diagrammatic representation of the method.
- 3.Describe the procedure and underlying principle of both solid-phase extraction and liquid-liquid extraction. Compare their advantages and limitations.
- 4. Analyze the role of extraction techniques in sample preparation and purification with pharmaceutical examples.
- 5.Write a detailed note on the different methods of Radioimmunoassay and their significance.
- 6.Evaluate the practical challenges and regulatory considerations in the use of RIA in modern labs.

# UNIT-V Hyphenated techniques-LC-MS/MS, GC-MS/MS, HPTLC-MS.

- 1. What does LC-MS/MS stand for?
- 2. Name the detector commonly used in LC-MS/MS.
- 3. What is the main advantage of using hyphenated techniques?
- 4.Expand HPTLC-MS.
- 5. Mention one application of GC-MS/MS.
- 6.Define the term "hyphenated technique" in analytical chemistry.

- 1.Briefly describe the working principle of LC-MS/MS.
- 2.Compare LC-MS/MS and GC-MS/MS with respect to sample types and applications.
- 3.Write a short note on the applications of HPTLC-MS in pharmaceutical analysis.
- 4.Explain the importance of tandem mass spectrometry (MS/MS) in modern analytical labs.
- 5. Discuss the components involved in GC-MS/MS instrumentation.
- 6.What are the advantages of coupling chromatography with mass spectrometry?

- 1. Discuss the principle, instrumentation, and applications of LC-MS/MS in pharmaceutical and biomedical analysis.
- 2. Explain in detail the working and applications of GC-MS/MS. Include sample preparation and detection process.
- 3. Describe the principle and significance of HPTLC-MS. How does it enhance traditional TLC techniques?
- 4. Compare and contrast LC-MS/MS, GC-MS/MS, and HPTLC-MS in terms of instrumentation, applications, and sensitivity.
- 5. Explain the concept of hyphenated techniques and discuss their advantages in complex sample analysis.
- 6. Analyze the role of tandem mass spectrometry (MS/MS) in structural elucidation and quantification of pharmaceutical compounds.