

Duration: 3 hours

Max. Marks: 75

Note:

1. Draw net labeled diagrams wherever applicable
2. Marks to the right indicate full marks

**I. Multiple choice questions**

**20M**

1. In a proton NMR spectrum, what does the term "chemical shift" refer to? **1M**
  - a. The separation between two peaks
  - b. The area under the peak
  - c. The position of a peak relative to a reference compound
  - d. The width of a peak at half its maximum height
2. In a DSC thermogram \_\_\_\_\_ is an example of exothermic reaction. **1M**
  - a. Crystallization
  - b. Melting point
  - c. Sublimation
  - d. Desolvation
3. Limit of quantification of analytical method is calculated by using the formula **1M**
  - a.  $10 \times$  standard deviation of intercept / Slope
  - b.  $3.3 \times$  standard deviation of intercept / Slope
  - c. standard deviation of intercept / Slope
  - d.  $10 \times$  standard deviation of intercept
4. Liquid liquid extraction is governed by \_\_\_\_\_ **1M**
  - a. Boyle's law
  - b. Ostwald dilution law
  - c. Nernst distribution law
  - d. Beer's law

5. In mass spectrometry, when electron impact (EI) ionization is used, the kinetic energy of the incident electrons is typically in the range of? 1M
- Millivolts
  - Kiloelectron volts
  - Megavolts
  - Microvolts
6. What does the acronym "MALDI" stand for in the context of mass spectrometry? 1M
- Molecular Analysis and Laser Detection Instrument
  - Matrix-Aided Laser Dissociation and Imaging
  - Matrix-Assisted Laser Desorption/Ionization
  - Mass Analyzing and Linear Detection Interface
7. \_\_\_\_\_ is the primary information obtained from an X-ray diffraction pattern. 1M
- The size of the crystal
  - The composition of the crystal
  - The arrangement of atoms in the crystal lattice
  - The crystal's color and transparency
8. \_\_\_\_\_ parameter used in Analytical method validation denotes closeness to the true value 1M
- LOQ
  - Accuracy
  - Selectivity
  - Precision
9. \_\_\_\_\_ is based on antigen antibody reaction. 1M
- Radioimmunoassay
  - X ray diffractometry
  - UV spectroscopy

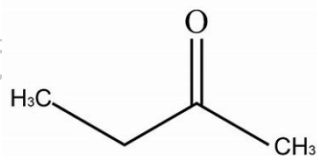
- d. IR spectroscopy
10. Which of the following interfaces of GCMS works on the principle of permeation of molecules through silicon rubber membrane? 1M
- a. Permeation interface
  - b. Molecular Jet separator
  - c. Capillary direct interface
  - d. Open split interface
11. In a proton NMR spectrum, what does the term "shielding" refer to? 1M
- a. The reduction in the magnetic field strength experienced by a nucleus due to nearby electron density
  - b. The enhancement of peak intensity by increasing the magnetic field strength
  - c. The broadening of peaks caused by rapid molecular motion
  - d. The conversion of time-domain data into frequency-domain data
12. In differential thermal analysis (DTA) \_\_\_\_\_ 1M
- a. The temperature differences between the sample and reference are measured as a function of temperature
  - b. The differences in heat flow into the reference and sample are measured as a function of temperature
  - c. The change in the mass of the sample is measured as a function of temperature
  - d. The glass transition is observed as a sharp peak
13. Holmium oxide solution is used for calibration of \_\_\_\_\_ of UV VIS 1M
- a. Resolution power
  - b. Stray light
  - c. Wavelength accuracy
  - d. Control of absorbance

14. Characteristic features of SPE include the following except \_\_\_\_\_ 1M
- a. Complete flexibility
  - b. Longer column lifetimes
  - c. Effective contaminant removal
  - d. Reduced sensitivity
15. What does the term "base peak" represent in a mass spectrum? 1M
- a. The peak corresponding to the molecular ion
  - b. The peak with the lowest mass-to-charge ratio ( $m/z$ )
  - c. The peak with the highest mass-to-charge ratio ( $m/z$ )
  - d. The most intense peak in the spectrum
16. In X-ray diffraction, what does the term "d-spacing" refer to? 1M
- a. The distance between the X-ray source and the sample
  - b. The distance between the X-ray detector and the sample
  - c. The distance between crystal lattice planes in a sample
  - d. The wavelength of the incident X-rays
17. \_\_\_\_\_ parameter in analytical method validation determines instrument performance by analysis of reference standard prior to running an analytical batch 1M
- a. System Suitability
  - b. Accuracy
  - c. Precision
  - d. Linearity
18. How many signals will be obtained in  $^1\text{H}$  NMR of n-propane 1M
- a. 1
  - b. 2
  - c. 3
  - d. 4

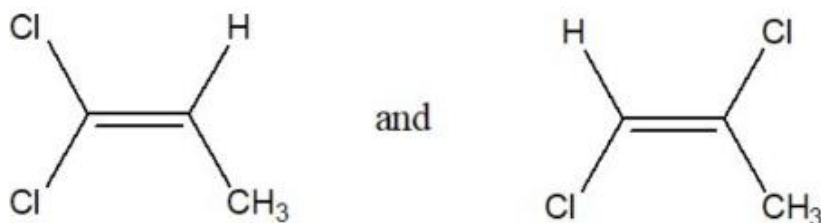
19. \_\_\_\_\_ involves measurement of a change in the weight of a substance as a function of temperature or time. 1M
- Thermogravimetry
  - Differential thermal analysis
  - Differential scanning calorimetry
  - IR spectroscopy
20. Which of the following is a study parameter for calibration of electronic weighing balance? 1M
- Flow rate accuracy
  - Drift
  - Resolution
  - Stray light

**II. Long answer questions (Attempt any two out of three) 20M**

1. a. An organic compound with molecular formula  $C_{10}H_{11}O_2Cl$  shows following IR: 1745, 1600 and 1500  $cm^{-1}$   
 $^1H$  NMR: 2.0 (s, 3H)  
 2.8 (t, 2H; J = 6Hz)  
 4.1 (t, 2H; J = 6Hz)  
 7.1 (d, 2H; J = 8Hz)  
 7.3 (d, 2H; J = 8Hz)  
 Deduce the structure and justify your answer 5M
- b. Give a detailed account of MALDI OR Chemical ionization as ionization technique in mass spectrometry 5M
2. a. Explain the concept of spin-spin coupling with an illustrative example. What is meant by 'J' value? 5M
- b. Explain the terms 'Mass spectrum' and 'Isotope peak' Depict one fragmentation pathway for the following compound? 5M



3. a. How will you distinguish between the following compounds using  $^1\text{H NMR}$ ? 5M



Justify your answer

- b. With the help of a neat labeled diagram explain the principle and working of time-of-flight mass analyzer. What is meant by mass to charge ratio 5M

**III. Short answer questions (Attempt any seven out of nine) 35M**

1. Enlist different parameters of analytical method validation. Explain linearity studies in detail 5M
2. Enlist different types of DSC instruments. Explain any one in detail. Describe a DSC thermogram 5M
3. Discuss principle involved in X-ray diffraction technique. Give the advantages and disadvantages of the technique. List any one pharmaceutical application 5M
4. Discuss the principle and instrumentation of DTA with suitable diagrams. Enlist applications of DTA technique 5M
5. Explain precision studies with respect to validation of analytical method 5M
6. Discuss calibration of FTIR in detail 5M
7. Discuss the various components and procedure for conduct of a radioimmunoassay and indicate the merits and demerits of 5M
8. Explain principle involved in LLE with suitable example. Give Applications of LLE. 5M
9. Discuss any one interface used in LCMS. Give applications of hyphenated technique 5M