**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.  Which of the following factors affects the chemical shift of a proton in an NMR spectrum?                                    1M

a.  Temperature

b.  Magnetic field strength

c.  Solvent polarity

d.  Pressure

2.  All of the following are types of DSC instruments except _____.                                     1M

a.  Heat flux

b.  Power compensated

c.  Modulated

d.  Hypo

3.  Lowest amount of analyte which can be quantified with accuracy using analytical method is termed as                                    1M

a.  LOQ

b.  LOD

c.  Selectivity

d.  Precision

4.  In liquid-liquid extraction, what does the term "partition coefficient" (K) represent?                                     1M

a.  The ratio of solute concentration in the extracting solvent to the initial concentration in the feed solution

b.  The ratio of solute concentration in the extracting solvent to its concentration in the raffinate

c.  The ratio of solute concentration in the raffinate to its concentration in the feed solution

d.    The ratio of solute concentration in the extracting solvent to its concentration in the extract phase

5.    Which of the following is a common matrix used in MALDI for the analysis    1M
of peptides and proteins?

a.    Trifluoroacetic acid

b.    Ammonium acetate

c.    2,5-dihydroxybenzoic acid

d.    Sodium chloride

6.    Which component of a mass spectrometer separates ions based on their mass-    1M
to-charge ratio (m/z)?

a.    Ionization source

b.    Mass Analyzer

c.    Detector

d.    Vacuum system

7.    Radioisotopes are used for the production of _____.    1M

a.    X-rays

b.    Radiofrequency waves

c.    UV rays

d.    IR radiation

8.    The precision parameter of an analytical method is usually expressed as    1M

a.    SEM

b.    Mean

c.    Coefficient of variation

d.    Percentage

9. In radioimmunoassay _____ is labeled with a radioactive isotope for detection.  1M

a. Antigen

b. Antibody

c. Substrate

d. Enzyme

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. Which unit is typically used to express chemical shift in proton NMR spectroscopy?  1M

a. Hertz (Hz)

b. Parts per million (ppm)

c. Tesla (T)

d. Degrees Celsius (°C)

12. DSC and DTA are based on _____  1M

a. Catalytic properties of enzyme

b. Elasticity of crystals

c. Enthalpy of substances

d. Line positions of phases

13. Which of the following is a study parameter for calibration of HPLC  1M

a. Resolution

b. Stray light

c. Flow rate accuracy

d. Vapor pressure

**56018** **Page 3 of 6**

14. Which of the following is an advantage of SPE over LLE? 1M

a. No emulsion formation

b. Less recovery

c. Less selectivity

d. Lesser plate numbers

15. What does the base peak represent in a mass spectrum? 1M

a. The most abundant ion in the spectrum

b. The ion with the highest m/z ratio

c. The ion with the lowest m/z ratio

d. The ion with the highest intensity

16. Radians is the unit of measurement used in the measurement of _____. 1M

a. X-ray diffraction angle

b. Absorption maxima

c. Radiofrequency

d. Vibrational frequency

17. _____is a measure of capacity of analytical method to remain 1M
unaffected by small, but deliberate variations in analytical method parameters
and provides an indication of its reliability during normal usage

a. Accuracy

b. Robustness

c. Linearity

d. Specificity

18. How many signals will be given by ethyl chloride in $^1$H NMR? 1M

a. 1

b. 2

c. 3

d. 4

19. The difference in heat flow into the sample and reference is measured in_____. 1M

a. DSC

b. DTA

c. TGA

d. IR spectroscopy

20. Potassium dichromate is used for calibration of _____ of UV-VIS spectrophotometer 1M

a. Resolution power

b. Stray light

c. Wavelength accuracy

d. Control of absorbance

## II. Long answer questions (Attempt <u>any two</u> out of three) 20M

1. a. A compound with molecular formula $C_6H_{12}O_2$ has the following spectral characteristics: 5M
IR (cm$^{-1}$): 3500, 2990, 1715
$^1$H NMR: $\delta 1.25$(s, 6H); 2.1(s, 3H); 2.6(s, 2H); 3.7(bs, $D_2O$ exchangeable)
Deduce the structure

b. Enlist any four methods of ionization in mass spectrometry. Give the principle, advantages and disadvantages of any one technique in detail 5M

2. a. Give the principle of NMR phenomena using proton as an example. Explain the terms 'Precessional frequency' and 'Gyromagnetic ratio' 5M

b. Depict two fragmentation pathways for the following compound (one fission and one rearrangement): 5M



**56018** **Page 5 of 6**
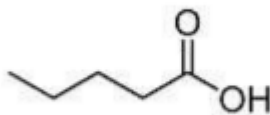
3. a. How will you distinguish between the following structures using $^1$H NMR? 5M



&

Justify your answer

b. Explain the term 'Mass spectrum'. With the help of a neat labeled diagram explain the principle and working of time-of-flight mass analyzer 5M

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

1. Define validation of analytical method and explain the following parameters 5M
   i. Accuracy ii. system Suitability

2. Explain principle and working of DSC instrument. Give any two applications of the technique 5M

3. Describe Miller's indices. Discuss principle involved in X-ray diffraction technique. Explain application of the method with the help of a case study 5M

4. Discuss the principle and instrumentation of DTA with suitable diagrams. Explain the DTA curve 5M

5. Give objectives of validation of analytical methods. Explain linearity studies with respect to validation of Analytical procedure 5M

6. Discuss calibration of uv-visible spectrophotometer in detail 5M

7. Explain the principle and methodology of Radioimmunoassay. Enlist merits and demerits of RIA 5M

8. Explain the principle and methodology of SPE. State advantages of SPE over LLE. Enlist sorbents used in the SPE 5M

9. Enlist different interfaces used in LCMS. Discuss any one in detail 5M

_____