(3 Hours) [Total Marks : 100

N.B. : (1) Attempt **all** questions from **Section I.**
(2) Attempt **any three** questions from **Section II.**
(3) Attempt **any two** questions from **Section III.**
(4) **Figures** to the **right** indicate **full marks.**

Section I

1. Attempt all questions. choose the correct option. **40**

(1) Increase in extent of conjugation of double bonded system results in _____.

(a) hyperchromic shift (b) hypochromic shift
(c) hypsochromic shift (d) bathochromic shift

(2) Presence of two filters in a photo fluorimeter.

(a) improves sensitivity (b) improves selectivity
(c) improves accuracy (d) improves precision

(3) In ^{1}H NMR spectroscopy, position of signals is denoted by _____.

(a) coupling constant (b) chemical shift
(c) precessional frequency (d) radio frequency

(4) Mc Laffarty rearrangement in mass spectrometry is shown by compounds having a double bond and _____.

(a) α H (b) β H
(c) γ H (d) δ H

(5) Number of signals observed in ^{13}C NMR spectrum of toluene is _____.

(a) 7 (b) 6
(c) 5 (d) 2

(6) In which technique temperature difference between sample and a reference is recorded as a function of time _____.

(a) Thermal gravimetric analysis
(b) Differential thermal analysis
(c) Thermometric titration
(d) Differential scanning thermometry

(7) Which wave number range is prescribed by the pharmacopoeia for IR measurements

(a) 60 - 208cm^{-1} (b) 206 - 560cm^{-1}
(c) 670 - 4000cm^{-1} (d) 3800 - 6000cm^{-1}

(8) 2N H_2SO_4 is _____.

(a) 1 M H_2SO_4 (b) 2 M H_2SO_4
(c) 5 M H_2SO_4 (d) 10 M H_2SO_4

(9) 10 ppm is _____.

(a) 1 µg/ml (b) 10 µg/ml
(c) 20 µg/ml (d) 0.5 µg/ml

(10) Bragg's equation is used in _____.

(a) UV spectroscopy (b) IR spectroscopy
(c) Raman spectroscopy (d) X ray diffraction

(11) We conclude that two means are significantly different if _____.

(a) the calculated t value is smaller than the tabulated t value
(b) the calculated t value is larger than the tabulated t value
(c) the calculated t value is equal to the tabulated t value
(d) the calculated t value is zero

(12) PAT stand for _____.

(a) Process analytical technology
(b) Procedure analytical test
(c) Permanent assessment test
(d) Process assessment test

(13) Additional laboratory testing for investigation of out of specification results involves.

(a) retesting (b) resampling
(c) retesting and resampling (d) neither retesting nor resampling

(14) For detection of polymorphs, the following technique can be used _____.

(a) UV spectroscopy (b) IR spectroscopy
(c) fluorimetry (d) Mass spectroscopy

(15) Precessional frequency depends on _____.

(a) the applied magnetic field
(b) the magnetic field experienced by the nucleus
(c) the difference between the applied field and the field experienced by the nucleus
(d) the magnetic field generated by the nucleus

(16) An example of a bulk property detector in HPLC is _____.

(a) Evaporative light scattering detector
(b) UV visible detector
(c) Fluorescence detector
(d) Phosphorescence detector

(17) Chiral chromatography involves separation of

(a) enantiomers (b) diastereomers
(c) geometrical isomers (d) radio isotopes

(18) Molecular ion peak is generally _____.

(a) peak with highest m/z value
(b) peak with highest relative intensity
(c) peak with lowest m/z value
(d) peak with lowest relative intensity

(19) Quadrupole analyser is used in _____.

(a) UV spectroscopy (b) IR spectroscopy
(c) NMR spectroscopy (d) Mass spectrometry

(20) Which of the following bands would have the lowest wave number? C = 0 stretch of _____.

(a) aliphatic amides (b) αβ unsaturated amides
(c) aliphatic ketones (d) αβ unsaturated ketones

(21) Head space analysis is used to _____.
- (a) determine moisture content
- (b) determine residual solvents
- (c) determine polymorphism
- (d) determine optical rotation

(22) For calibration of absorbance in UV spectroscopy, the material/s used is / are _____.
- (a) holmium filter
- (b) polystyrene
- (c) potassium dichromate
- (d) toluene and hexane

(23) In radial paper chromatography sample is placed _____.
- (a) near the outer surface of the paper
- (b) at the centre of the paper
- (c) at the bottom of the paper
- (d) at the top of the paper

(24) C = C stretch for an alkene is observed at _____.
- (a) $1620cm^{-1}$
- (b) $1720cm^{-1}$
- (c) $1520cm^{-1}$
- (d) $1820cm^{-1}$

(25) In normal phase chromatography _____.
- (a) stationary phase is polar and mobile phase is polar
- (b) stationary phase is non-polar and mobile phase is non-polar
- (c) stationary phase is polar and mobile phase is non-polar
- (d) stationary phase is non-polar and mobile phase is polar

(26) ELISA means _____.
- (a) Enzyme Linked Immuno Sorbent Assay
- (b) Enzyme Linked Immunological Serum Assay
- (c) Enzyme Linked Immunomodulatory Serum Assay
- (d) Enzyme Linked Immunity Serum Assay

(27) The number of disintegration per second for one curie is

(a) 1 (b) 7.3×10^{10}

(c) 3.7×10^{10} (d) 1×10^{10}

(28) The chemical shift range for ^{13}C NMR signal of carbonyl group is _____.

(a) 10 - 30 ppm (b) 50 - 60 ppm

(c) 100 - 120 ppm (d) 160 - 240 ppm

(29) In limit of detection, signal to noise ratio is _____.

(a) 2 : 1 (b) 3 : 1

(c) 8 : 1 (d) 10 : 1

(30) Carboxylic acid group is an example of _____.

(a) weak cation exchange group

(b) weak anion exchange group

(c) strong cation exchange group

(d) weak anion exchange group

(31) Wall coated open tubular columns are used in _____.

(a) HPLC (b) Ion exchange chromatography

(c) Gas chromatography (d) Gel permeation chromatography

(32) Resolution is affected by _____.

(a) only difference in retention times of the chromatographic peaks

(b) only width of the chromatographic peaks

(c) both difference in retention times and width of the chromatographic peaks

(d) neither difference in retention times nor width of the chromatographic peaks

(33) Heptane sulfonic acid is an example of _____.

(a) ion exchange reagent (b) ion pair reagent

(c) ion dissolving reagent (d) ion chelating reagent

(34) For supercritical fluid, which statements are true _____.

(A) Temperature and pressure go beyond critical point

(B) It is able to solubilise both polar and non-polar molecules

(C) It is able to solubilise only polar molecules

(D) Temperature and pressure do not go beyond critical point

(i) Statement A and B are true

(ii) Statement A and C are true

(iii) Statement D and B are true

(iv) Statement D and C are true

(35) The ICH guideline for Impurities : Guideline for residual solvents is given in _____.

(a) Q3A (R2) (b) Q3B (R2)

(c) Q3C (R5) (d) Q3D

(36) If a chromatographic peak starts at 5.1 minutes ends at 5.5 minutes and has a maximum at 5.3 minutes, the number of theoretical plates (N) are _____.

(a) 2800 (b) 2804

(c) 2809 (d) 2815

(37) Capacity factor of a component in chromatography is _____.

(a) Adjusted retention time of solute divided by dead time

(b) Retention time of solute divided by dead time

(c) Retention time of more retained component to less retained component

(d) Adjusted retention time of solute divided by retention time of solute

(38) In thermogravimetric analysis the property measured is _____.

(a) change in weight (b) change in temperature

(c) rate of change of weight (d) rate of change of temperature

(39) The order of energy used in the techniques is _____.

(a) UV > IR > NMR (b) IR > UV > NMR

(c) NMR > UV > IR (d) UV > NMR > IR

(40) A commonly used reagent gas in chemical ionisation is _____.

(a) Nitrogen (b) Oxygen

(c) Carbondioxide (d) Methane

Section II

2. Attempt **any three** questions : (10 x 3 = 30) **30**

(1) Explain the following terms :

(a) Gradient elution

(b) Radiochemical purity

(c) Working standard

(d) Correlation coefficient

(2) Enlist the problems faced by pharmaceutical analyst during development of analytical method for the assay of low dosage drugs from formulations. Explain in detail trouble shooting for the same keeping in view green analytical chemistry.

(3) Explain the term PAT. Give suitable examples for the same.

(4) Discuss current analytical aspects in quality control of herbals.

(5) Calculate number of theoretical plates for A and B and resolution between A and B for following data :

HPLC column length - 25 cm

	Retention time (min)	**Width of peak at base (min)**
Unretained compound	1.5	0.3
A	4	1.3
B	7	1.8

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Section III

3. Attempt **any two** questions : **30**

(1) In bioanalytical studies LC-MS is the method of choice. Comment on the above statement and justify your answer. Write a note on LC-MS method development for analysis of drug and metabolites from a biological matrix.

(2) Documentation is very important in research. Discuss the importance of proper documentation in research taking suitable examples.

(3) Discuss the importance of forced degradation studies in development of stability indicating assay method. How will you conclude that the method developed is stability indicating.

(4) What are the analytical considerations for dissolution studies for new drug formulations. Write a note on current trends in dissolution testing of solid dosage forms.
