MODERN INSTRUMENTAL METHODS OF ANALYSIS
(CHEN 4131)

Time Allotted : 2½ hrs **Full Marks : 60**

Figures out of the right margin indicate full marks.

Candidates are required to answer Group A and any 4 (four) from Group B to E, taking one from each group.

Candidates are required to give answer in their own words as far as practicable.

Group – A

1. Answer any twelve: **12 × 1 = 12**

Choose the correct alternative for the following

(i) If I_0 is the incident radiation and I is the transmitted radiation, absorbance A is expressed as
(a) log I (b) log (I_0/I) (c) I_0/I (d) log (I/I_0)

(ii) A solution containing 0% hexadecane shows an absorbance of 0.002 at 3.41 μm and a solution with 4 % hexadecane shows absorbance of 0.103 at the same wavelength. The corrected absorbance for the 4% hexadecane solution is
(a) 0.103 (b) 0.002 (c) 0.101 (d) None of these.

(iii) If percentage transmittance is 50, absorbance is
(a) 0.3 (b) 0.5 (c) 0.1 (d) 0.8.

(iv) The type of flame used in a flame atomizer is
(a) nitrous oxide acetylene flame (b) air acetylene flame
(c) air hydrogen flame (d) Both (a) and (b).

(v) Grotrian diagrams are plots of
(a) energy of atoms versus possible atomic energy levels
(b) energy of molecules versus possible atomic energy levels
(c) energy of atoms versus wavelength
(d) Both (a) and (b).

(vi) Photodiode arrays are a type of
(a) radiation source (b) detector
(c) monochromator (d) autosampler.

(vii) The law relating wavelength and angle of diffraction in X-ray diffraction is known as
(a) Beer's law (b) Bragg's law (c) Snell's law (d) Moseley's law.

(viii) Infrared radiation causes a change in
(a) Translational modes in molecules (b) Vibrational modes in molecules
(c) Rotational modes in molecules (d) Electronic transition

(ix) Organic nitrogen compounds are detected using
(a) Flame photometric detector (b) Flame thermionic detector
(c) Electron capture detector (d) RI detector.

(x) Chromatography operates on the principle of
(a) filtration (b) sedimentation (c) partitioning (d) absorption.

Fill in the blanks with the correct word

(xi) The detector that operates on the principle of change in electrolytic conductivity due tpo the presence of halogen, sulphur or nitrogen atoms in the analyte is known as _____.

(xii) The m/z ratio in mass spectrometry can be expressed as _____.

(xiii) The phenomenon of reduction in absorbance of metal atoms due to presence of thermally stable compound is known as _____.

(xiv) Selenium can be best detected using _____.

(xv) Absorption of IR radiation results in a change in the _____ moment of the molecule/bond.

Group - B

2. (a) Discuss the analytical applications of UV-Vis spectrophotometer. *[(CO3)(Remember/LOCQ)]*
(b) Explain the effects of solvent on UV spectra. *[(CO1)(Remember/LOCQ)]*
(c) If a wavelength of 653 nm is absorbed by glass, calculate the difference in energy between the ground and excited state of glass. The value of Plancks constant is 6.626×10^{-34} J s and speed of light is 3×10^{8} m/s. *[(CO1)(Apply/IOCQ)]*

5 + 4 + 3 = 12

3. (a) Discuss the principle of operation of UV/Vis spectroscopy. Name the different components of a UV/Vis spectrophotometer. *[(CO2)(Remember/LOCQ)]*
(b) Name the detector used in UV/Vis spectrophotometer. Explain the working principle of the detector with diagram. *[(CO2)(Remember/LOCQ)]*

6 + 6 = 12

Group - C

4. (a) Discuss ways to eliminate different types of interferences in AAS. *[(CO3)(Remember/LOCQ)]*
(b) Explain the methods of analysis of samples using AAS. *[(CO4)(Remember/LOCQ)]*

6 + 6 = 12

5. (a) Discuss the working principle of a spark excitation source with diagram. *[(CO3)(Analyse/LOCQ)]*
(b) Discuss the working principle of an AFS. State the applications of AFS. *[(CO4)(Understand/LOCQ)]*
(c) Discuss the analytical applications of flame OES. *[(CO3)(Analyse/IOCQ)]*

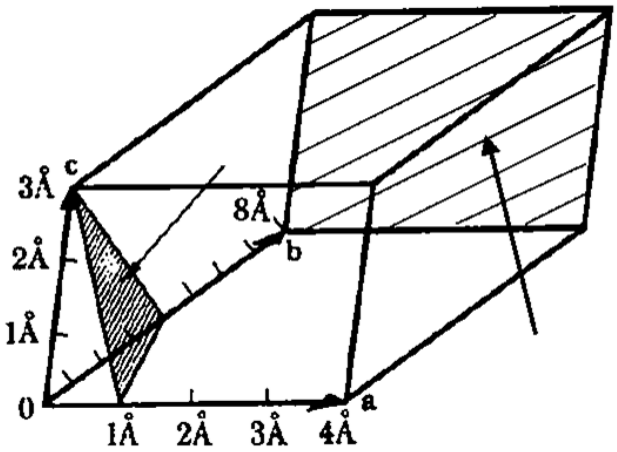
5 + 4 + 3 = 12

Group - D

6. (a) State and explain Bragg's law. *[(CO1)(Remember/LOCQ)]*

(b) Explain the concept of Miller indices. *[(CO1)(Remember/LOCQ)]*

(c) Compute the Miller indices for the shaded planes. *[(CO3)(Apply/HOCQ)]*



4 + 4 + 4 = 12

7. (a) The mass spectra of butanol, 2-butanol and 2 methyl-2-propanol are shown below. Molecular weight of the three isomers is 74. Interpret the results. Explain the significance of m/z. What does the value 100 corresponding to m/z =31 for the different isomers of butanol represent? *[(CO4)(Apply/HOCQ)]*

m/z	15	27	29	31	33	39	42	43	45	55	56	57	59	60	74
Butanol	8.4	50.9	29.9	100	8.5	15.6	32.4	61.4	6.6	12.3	99.9	6.7	0.3	0	0.8
2-butanol	6.8	15.9	13.9	20.31	0	3.4	1.7	9.8	100	2.0	1.0	2.7	17.7	0.7	0.3
2-methyl-2-propanol	13.3	9.9	12.7	35.5	0	7.7	3.3	14.5	0.6	1.6	1.5	9.0	100	3.2	0

(b) Calculate the particle size using Scherrer equation with the following information obtained from XRD of $CoFe_2O_4$ sample, wavelength of copper Kα line 1.5406 A, Bragg's angle of diffraction 36.23, Miller index (311) and FWHM 0.4 degree. *[(CO4)(Analyse/HOCQ)]*

(3 + 2 + 2) + 5 = 12

Group - E

8. (a) Explain the working principle of an HPLC pump with diagram. *[(CO4)(Remember/LOCQ)]*

(b) For a capillary GC column, 30 m long, with a diameter of 0.25 mm, operated at a constant temperature of 200°C under a helium pressure of 20 psi above atmospheric pressure, resulting in an average linear flow rate of 30.4 cm/s, the following data is given on 5 analyte peaks and one standard peak.

Peak (s)	t_r (min)	w_b (s)	w_b (1/2)(s)
1	3.70	5.13	3.02
2	4.00	5.54	3.26
3	4.10	5.68	3.34
4	4.30	5.96	3.51
5	4.35	6.03	3.55
IS	8	11.09	6.52

The number of theoretical plates is 30000.
(i) Compute HETP and comment if the column is efficient
(ii) Calculate the retention factor and resolution for each of the peaks.

[(CO4)(Apply/HOCQ)]

6 + (2 + 4) = 12

9. (a) In the following table, data for peaks 1,2 with internal standard IS1 from standard curve runs is provided. The table also contains data for unknown peaks A, B, C eluting with similar retention time alongwith internal standard IS2. Compute the RRT values and draw conclusions based on these values.

Peak	1	A	2	B	C	IS1	IS2
RT (min)	4	4.01	4.10	4.12	4.24	8	8.2

[(CO3)(Analyse/HOCQ)]

(b) Discuss the basic principle of chromatographic separation. *[(CO4)(Remember/LOCQ)]*

6 + 6 = 12

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	64.58	6.25	29.16