

**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) The absorption of UV radiation results in
 (a) rotational transition in molecules (b) vibrational transition in molecules
 (c) electronic transition in molecules (d) Both (b) and (c)
- (ii) The relation between absorbance A and transmittance T is given as
 (a) $T = -\log A$ (b) $A = T$
 (c) $A = 1 - T$ (d) $A = -\log T$
- (iii) An example of photon detector is
 (a) Thermocouple (b) Golay detector
 (c) Refractive index detector (d) Photomultiplier tube
- (iv) The detector used in AAS is
 (a) Photomultiplier tube (b) Thermistor
 (c) Photodiode array (d) Both (a) and (c)
- (v) The efficiency of atomization by air-acetylene flame is highest for
 (a) Aluminium (b) Barium
 (c) Gallium (d) Copper
- (vi) The most suitable technique for mercury detection is
 (a) AAS (b) AFS
 (c) AES (d) UV-Vis spectroscopy
- (vii) Spin angular momentum is represented as
 (a) $\frac{h}{2\pi} \sqrt{I(I+1)}$ (b) $\frac{h}{2\pi \sqrt{I(I+1)}}$
 (c) $\frac{2\pi}{h} \sqrt{I(I+1)}$ (d) $\frac{h}{2\pi} I$
- (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) Stationary phase in HPLC is composed of
 (a) n-C₁₈ hydrocarbon (b) ceramic
 (c) polymer (d) metal
- (x) The ratio of concentrations of components in the stationary phase to mobile phase is known as
 (a) Retention factor (b) Equilibrium partition ratio
 (c) Partitioning volume phase ratio (d) Absorption retention time

Fill in the blanks with the correct word

- (xi) The relation between concentration of an analyte and signal in the method of internal standard calibration can be expressed as _____.
- (xii) The spectroscopic technique that is based on scattering of light is known as _____.
- (xiii) Example of an electrothermal atomizer is _____.
- (xiv) EDXRF instruments can determine elements from _____.
- (xv) Absorption of IR radiation results in a change in the _____ moment of the molecule/bond.

Group - B

2. (a) State and explain Beer's law. [[CO1](Understand/LOCQ)]
 (b) Discuss the advantages of instrumental methods over classical methods of analysis. [[CO1](Remember/LOCQ)]
 (c) Distinguish between absorption and emission spectra using schematic diagram. Classify the spectroscopic techniques that use UV/visible radiation sources. [[CO1](Analyse/IOCQ)]

3 + 4 + (3 + 2) = 12

3. (a) The presence of lead in water was determined using ICP-MS. Two lead calibration standards were prepared, one containing 10 ppb lead and other containing 10 ppb lead and 20 ppb bismuth as internal standard. The data for calibration using internal standard is shown in Table 1. An unknown water sample to which 20 ppb Bismuth is added as internal standard is subjected to ICP-MS analysis and the results are shown in Table 2. Calculate the unknown lead concentration at various time points and compute the percentage error in results obtained.

Table 1 Calibration with internal standards

Time of measurement	8AM	10AM	1PM	3PM
Signal counts 10 ppb Pb	12050	12100	30000	15750
Signal counts 20 ppb Bi	60000	60080	149200	78400

Table 2 Unknown lead sample in water containing internal standard

Time of measurement	8AM	10AM	1PM	3PM
Signal counts for Pb	6028	6063	15010	7789
Signal counts for Bi	60010	60075	149206	78398

[[CO1](Apply/HOCQ)]

- (b) Distinguish between nephelometry and turbidimetry using a schematic diagram. [[CO2](Analyse/IOCQ)]
7 + 5 = 12

Group - C

4. (a) Explain what is meant by spectral linewidth. Discuss the different types of linewidth broadening observed in AAS. [[CO3](Analyse/IOCQ)]
(b) Discuss the working principle of a flame atomizer in AAS with a neat flow diagram. [[CO4](Remember/LOCQ)]
6 + 6 = 12
5. (a) Write the Maxwell-Boltzmann equation and explain each term. [[CO1](Analyse/HOCQ)]
(b) "Atomic absorption with flame atomizers can not be used for the determination of non-metals. Justify the statement. [[CO4](Remember/LOCQ)]
(c) Sketch and explain the Grotrian diagram of sodium. Explain chemical interferences in AAS. [[CO1](Apply/IOCQ)]
3 + 3 + (3 + 3) = 12

Group - D

6. (a) Explain the working principle of a Michelson interferometer with a diagram. [[CO4](Understand/LOCQ)]
(b) Explain the basic difference in the working principle of EDXRF and WDXRF. State the applications of quantitative XRF. Write the equation relating concentration of an element with intensity of the signal detected in an XRF. [[CO4](Analyse/IOCQ)]
6 + (3 + 2 + 1) = 12
7. (a) Define magnetogyric ratio in NMR. Sketch the energy level of a nucleus with spin quantum number $\frac{1}{2}$ in the presence of a magnetic field and derive the Larmor equation. [[CO3](Analyse/IOCQ)]
(b) Explain the term 60 MHz proton NMR. [[CO4](Remember/LOCQ)]
(c) Discuss the analytical applications of NMR. [[CO4](Apply/IOCQ)]
(2 + 4) + 2 + 4 = 12

Group - E

8. (a) Define (i) partition ratio (ii) peak resolution and (ii) retention factor in chromatography. [[CO4](Understand/LOCQ)]
(b) "Number of theoretical plates is a measure of efficiency of a chromatographic column." Justify. [[CO4](Analyse/IOCQ)]
(c) State and explain van Deemter equation. [[CO4](Remember/LOCQ)]
4 + 4 + 4 = 12
9. (a) Explain the working principle of SPME with diagram. [[CO3](Understand/LOCQ)]

- (b) Draw the chromatogram of a single retained component and explain the important information that can be extracted from the chromatogram. *[(CO4)(Apply/HOCQ)]*
- (c) Define the following explaining their significance: (i) eluent strength (ii) polarity index. *[(CO3)(Apply/IOCQ)]*
- 4 + 4 + 4 = 12**
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Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	37.5	47.91	14.59