2023

CHEMISTRY — HONOURS

Paper: DSE-A-4

(Analytical Methods in Chemistry)

Full Marks: 50

The figures in the margin indicate full marks.

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

1. Answer any ten questions:

 1×10

- (a) What is Interferogram?
- (b) What is percentage transmittance (%T)?
- (c) Mention one functional group present each in cation exchange and anion exchange resin.
- (d) Why KBr is mostly used to prepare pellet in FTIR spectroscopy?
- (e) How can you represent calomel electrode?
- (f) What is liquid-junction potential?
- (g) Give two examples of two commonly used developers in paper chromatography.
- (h) Draw the pH metric titration curve for $H_2C_2O_4$ with NaOH.
- (i) Mention a factor that affects the end point sharpness in an acid-base titration?
- (j) How can the hollow cathode lamp be modified for multi-elemental analysis in AAS (atomic absorption spectrometer)?
- (k) Mention the S.I. unit of specific conductance and equivalence conductance.
- (l) Define specific rotation of an optically active substance. Mention its unit in CGS system.

Answer any eight questions.

- 2. (a) Mention two possible interferences in AAS and suggest methods to remove them.
 - (b) Mention the components present in a single beam AAS. What is the role of chopper?

3+2

- 3. (a) What is stationary and mobile phase in thin layer chromatography (TLC)? R_f values of three amino acids A1, A2 and A3 are 0.18, 0.40 and 0.66 respectively. Which one of these amino acids in their TLC separation will occur on the top and which one at the bottom? Explain.
 - (b) Which one is advantageous between atomic absorption and atomic emission spectroscopy and why? 3+2
- **4.** (a) Draw a schematic diagram of a double beam ultraviolet (UV-vis) spectrometer and describe its source and detector.
 - (b) What is meant by temperature programming in GC?

3+2

Please Turn Over

- 5. (a) Using solvent extraction method, how can you separate two organic compounds from their mixture? Is it useful for the separation of inorganic compound? Explain.
 - (b) What types of species can be separated by HPLC but not by GC?

3+2

- 6. (a) Discuss the principle of estimation of sodium ion in water sample by atomic emission spectrometry.
 - (b) Why is TMS a good reference compound in NMR spectroscopy?

3+2

- 7. (a) How can you separate a solution mixture containing $[Ni(H_2O)_6]^{2+}$ and $[Cr(nta)(H_2O)_2]$ with the help of ion exchange resin? (nta = nitrilotriacetate ion)
 - (b) Mention two operating systems and two programming languages which are used for analysing experimental data. 3+2
- **8.** (a) Mention the most common feature in furnace atmosphere employed in thermogravimetric (TGA) analysis. Also explain the role of humidified air used as furnace atmosphere.
 - (b) The extinction coefficient of $[Co(en)_2Br_2]^+$ is 40 M⁻¹cm⁻¹ at 650 nm. Calculate the % transmission for a 1 cm cell filled with 0.01 (M) solution.
- **9.** (a) Why is it necessary to calibrate the glass electrode before performing pH metric analysis of a given solution? Why should the glass electrode be preserved in saturated KCl solution?
 - (b) Predict the specific rotation of a mixture of 30% of (-)-2-bromobutane having $[\alpha]_D = -23.13^\circ$ and 70% of (+)-enantiomer.
- 10. (a) Write the method of estimation of Ca⁺² and Mg⁺² in a mixture using ammonium oxalate in thermogravimetric (TGA) method and briefly discuss the mass% vs. Temperature TG plot.
 - (b) 'Chromatography is a process of separation of substances by differential migration.' Explain.

3+2

- 11. (a) Mention how the analyses of different metals are influenced by the nature of flame in flame emission spectrometry (FES).
 - (b) Describe the HPLC technique to separate a racemic mixture of an ester with excellent resolution.
- **12.** (a) Comment on how the following factors can be controlled to attain effective result in conductometric precipitation titrations:
 - (i) Solubility of the precipitate during titration
 - (ii) Rate of precipitation
 - (b) In the IR spectrum of CO there is an intense band at 2143 cm⁻¹. Calculate the fundamental vibration frequency of CO and the force constant.