

2021

**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.*

Answer **question number 1** and **any eight** from the rest.

1. Answer **any ten** questions: 1×10
- (a) What do you mean by distribution coefficient in solvent extraction technique?
  - (b) What are the largest and smallest  $R_f$  values possible in chromatographic technique?
  - (c) Write an example of chiral shift reagent and mention its use.
  - (d) Draw the nature of curves expected for the titration of oxalic acid vs sodium hydroxide by pH-metric method.
  - (e) What is molar absorptivity?
  - (f) What is a charge coupled device?
  - (g) Among cellulose and silica gel, which one is better absorbent for the separation of metal ions in TLC technique and why?
  - (h) The IR vibrational frequency of tri atomic molecule like  $H_2O$  is found to be higher than the deuterium substituted molecule ( $D_2O$ )—Comment.
  - (i) Give one example each of chemical components used as stationary phase in TLC and Column chromatography.
  - (j) What types of fuels and oxidants are used in flame atomic absorption spectrometry and flame atomic emission spectrometry?
  - (k) Mention two softwares used in computer applications to plot a graph utilizing the experimental data.
  - (l) What are the deviations of Beer's law in spectrophotometry?
2. (a) A compound of formula weight 280 absorbed 69% of the radiation at certain wavelength in a 2 cm cell at a concentration of  $18.0 \mu\text{g/L}$ . Calculate the molar absorptivity of the compound. 3
- (b) State Beer-Lambert's law and give mathematical expression for it. 2

**Please Turn Over**

3. (a) Discuss the nature of curves expected with proper explanation during the titration of a mixture of 0.02 (M) sulfuric acid and 0.02(M) acetic acid vs 0.02(M) sodium hydroxide in a conductometric method. 3
- (b) What is a salt bridge? Discuss its utility. 2
4. (a) In a particular thin layer chromatographic (TLC) separation, the  $R_f$  value of unknown compound is 0.809. The fronts due to compounds A, B and C are 23.0, 29.0 and 32.0 cm respectively with the solvent front as 34.0 cm. Identify the unknown compound among A, B and C accumulate from top to bottom in the TLC stationary phase with proper explanation. 3
- (b) Absorptivity of ions will depend entirely on the relative charges and ionic radii of the two ions in ion exchange chromatography. — Explain. 2
5. (a) Distribution coefficient of chloride and bromide ions are 29 and 65 respectively. Show that their separation by ion exchange chromatography is favorable. 3
- (b) Write the criteria of solvent for the extraction of components in liquid-liquid solvent extraction method. 2
6. (a) Write the method of estimation of  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  in a mixture using ammonium oxalates in thermo gravimetric (TGA) method. 3
- (b) Briefly discuss the variation of mass% with temperature in a TG plot. 2
7. (a) Write down the different types of interference encountered during analysis with atomic absorption spectrometry. 3
- (b) For a diatomic molecule like hydrofluoric acid (HF) the IR vibrational frequency of this molecule is found at  $845\text{ cm}^{-1}$ . Whether any change in the region of IR vibrational frequency observed if the hydrogen atom of this molecule is substituted with deuterium? Explain your observation. 2
8. (a) State the principle of determination of solubility product of AgCl by potentiometric titration of standard KCl solution against  $\text{AgNO}_3$  solution. 3
- (b) An optically active 0.2 (M) concentrated solution of compound R has an observed rotation in a 10 cm cell of (+)  $0.4^\circ$ . The molecular weight of the compound is 150; calculate the specific rotation of R. 2
9. (a) Compare reverse phase operation and normal phase operation in HPLC. 3
- (b) What is the use of pre-heater in gas chromatography? 2
10. (a) Discuss the principle of estimation of sodium ion in water sample by atomic emission spectrometry. 3
- (b) Mention two important roles of computers in the instrumental methods of analysis. 2

11. (a) How can you measure  $E^\circ_{\text{Fe}^{3+}/\text{Fe}^{2+}}$  when Mohr's salt solution is titrated potentiometrically using reference saturated calomel electrode. 3
- (b) In conductometric titration; it is preferable to titrate  $\text{AgNO}_3$  with  $\text{LiCl}$  solution rather than with  $\text{HCl}$  solution. Explain. 2
12. (a) 'Multiple extraction is better than single extraction with the same volume of the solvent.'— Discuss. 3
- (b) What is chelating ion exchange resin? 2
-