## 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:

 $1 \times 10$ 

- (a) What do you mean by distribution coefficient in solvent extraction technique?
- (b) What are the largest and smallest R<sub>f</sub> 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.
- (1) What are the deviations of Beer's law in spectrophotometry?
- (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 μg/L. Calculate the molar absorptivity of the compound.
  - (b) State Beer-Lambert's law and give mathematical expression for it.

2

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.
	(b)	What is a salt bridge? Discuss its utility.
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.
	(b)	Absorptivity of ions will depend entirely on the relative charges and ionic radii of the two ions in ion exchange chromatography. — Explain.
5.	(a)	Distribution coefficient of chloride and bromide ions are 29 and 65 respectively. Show that their separation by ion exchange chromatography is favorable.
	(b)	Write the criteria of solvent for the extraction of components in liquid-liquid solvent extraction method.
6.	(a)	Write the method of estimation of $Ca^{2+}$ and $Mg^{2+}$ in a mixture using ammonium oxalates in thermogravimetric (TGA) method.
	(b)	Briefly discuss the variation of mass% with temperature in a TG plot.
7.	(a)	Write down the different types of interference encountered during analysis with atomic absorption spectrometry.
	(b)	For a diatomic molecule like hydrofluoric acid (HF) the IR vibrational frequency of this molecule is found at 845 cm <sup>-1</sup> . 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.
8.	(a)	State the principle of determination of solubility product of AgCl by potentiometric titration of standard KCl solution against AgNO <sub>3</sub> solution.
	(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.
9.	(a)	Compare reverse phase operation and normal phase operation in HPLC.

10. (a) Discuss the principle of estimation of sodium ion in water sample by atomic emission

(b) Mention two important roles of computers in the instrumental methods of analysis.

2

32

(b) What is the use of pre-heater in gas chromatography?

spectrometry.

(2)

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2

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