ST. JOSEPH'S COLLEGE (AUTONOMOUS), BANGALORE-27
M. Sc. CHEMISTRY – II SEMESTER
SEMESTER EXAMINATION: APRIL – 2022
(Examination conducted in July 2022)
CH/OCH 8521: SEPARATION TECHNIQUES

Time- 2 ½ hrs.   **Max Marks-70**

This paper contains 3 printed pages and 3 parts and 17 questions.
Answer ALL the parts.

PART A

Answer any 6 out of 8 questions. Each question carries 2 marks.   [6 x 2 = 12]

1. Write the reaction of dithizone chelating agents with dipositive metal in the solvent extraction process.

2. Sketch the fronting and tailing asymmetric peaks.

3. What is the different between gradient elution and isocratic elution?

4. The distribution co-efficient for X between CCl_4 and H_2O is 19. If 50 ml of an aqueous solution of 0.25M is contact with 100ml of CCl_4, calculate the percentage of X remaining in the water.

5. Write any three properties of supercritical fluid.

6. What is a spacer arm?

7. Discus Prikle brush phases.

8. List any three applications of thin layer chromatography.

PART B

Answer any 4 out of 6 questions. Each question carries 12 marks.   [4 x 12 = 48]

9. a) Show that distribution ratio D depends on the concentration of weak acid HA when weak acid HA dimerize in organic phase.

    b) A 250 mg sample of organic compound (molecular weight = 84) is dissolved in 200 mL of water. The aqueous phase is contacted with 50 mL of benzene and allowed to reach equilibrium. Analysis of the aqueous phase now shows that it contains only 63 mg of the organic compound. Calculate K_D.

    c) An organic acid $K_a = 2 \times 10^{-6}$ has a distribution coefficient of 18.0 between water and methyl isobutyl ketone. If 100 mL of an aqueous solution buffered at pH 6.00 is extracted three times with 50 mL of methyl isobutyl ketone, calculate the percentage remaining in the aqueous phase.

10. a) What is meant by Scaling up? Explain the scaling up rules.

    b) Explain the Van Deemter equation for plate height.

    c) A chromatogram with ideal Gaussian bands has t_r = 9.0 min and $w_{1/2}$ = 2.0 min.

        (a) How many theoretical plates are present?

        (b) Find the plate height if the column is 10 cm long.

11. a) Explain the different types of open tubular columns.

    b) What are the advantages of temperature and pressure programming in gas chromatography?

    c) Explain the sample preparation in gas chromatography using fused silica fiber coated syringe with a suitable diagram.

12. a) Discuss the characteristics of evaporative light scattering detector used in liquid chromatography.

    b) Discuss the characteristic features of monolithic column.

    c) What is the difference between dead volume and dwell volume? How do each of these volumes affect a chromatogram?

13. a) Discuss the elution process between normal phase and reversed phase chromatography.

    b) Explain the Donnan equilibrium and give its application.

    c) Draw a schematic diagram and explain the suppressed-ion cation chromatography.

14. a) Explain the Ni^{2+} immobilized metal affinity chromatography.

    b) Discuss the preparation of thin layer plates in thin layer chromatography.

    c) Describe the ascending and descending paper chromatography.

PART C

Answer any 2 out of 3 questions. Each question carries 5 marks.   **[2 x 5 = 10]**

15. a) Why does a thermal conductivity detector respond to all analytes except the carrier gas? Why isn't the flame ionization detector universal?

    b) An unknown compound was co-chromatographed with heptane and decane. The adjusted retention times were heptane,12.6 min; decane, 22.9 min; unknown, 20.0 min. The retention indexes for heptane and decane are 700 and 1000, respectively. Find the retention index for the unknown.   (2+3)

16. a) Nonpolar aromatic compounds were separated by HPLC on an octadecyl (C18) bonded phase. The eluent was 65 vol% methanol in water. How would the retention times be affected if 90% methanol were used instead?

(b) Octanoic acid and 1-aminooctane were passed through the same column as above (16 a), using an eluent of 20% methanol/80% buffer (pH 3.0). State which compound is expected to be eluted first and why?

i) $CH_3CH_2CH_2CH_2CH_2CH_2CH_2CO_2H$ ii) $CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2NH_2$

Octanoic acid   1-Aminooctane

(2+3)

17. a) After optimization of an isocratic elution with several solvents, the resolution of two peaks is 1.2. How you might improve the resolution without changing solvents or the kind of stationary phase?

b) Analyze the thin layer chromatogram in 80% methanol as mobile phase, with compound A- n- Hexane, B- Diol, C- toluene.

i)   Calculate the R_f values for all components.
ii) Which component has the highest affinity for mobile phase? Explain.
iii) Which component has the highest affinity for stationary phase? Explain.

(2+3)

--------------------End of questions-------------------