

Registration Number:

Date & session:



ST. JOSEPH'S UNIVERSITY, BENGALURU -27
M.Sc (CHEMISTRY) – II SEMESTER
SEMESTER EXAMINATION: APRIL 2023
(Examination conducted in May 2023)
CH 8521-SEPARATION TECHNIQUES
(For current batch students only)

Time: 2 Hours

Max Marks: 50

This paper contains TWO printed pages and THREE parts.

PART-A

Answer any EIGHT of the following:

[8 x 2 =16]

1. Draw an isotherm for an overcrowded column. Explain the resulting band shape.
2. A sample containing 4 analytes are separated using polar column in GC. The analytes and their respective boiling points are given below. Plot a chromatogram showing the order of elution.

Analyte	Boiling point (°C)
Pentane	36
Butanol	117
Propanol	97
Octane	126

3. Which method of sample injection in GC would you choose in following cases:
 - i) Very dilute sample
 - ii) Sample containing thermally sensitive compounds
4. Explain the following: i) dwell volume ii) dwell time.
5. List any two properties of supercritical fluids, which are of importance in chromatography.
6. Write the elution equation used in molecular exclusion chromatography. Explain the terms.
7. Explain the need of the following:
 - i) Spacer arm in affinity chromatography
 - ii) Derivatization in chromatography
8. Explain i) isocratic elution ii) gradient elution.
9. Define the term $pH_{1/2}$, used in solvent extraction.
10. Write any two methods used for the preparation of thin layer plates in TLC.

PART-B

Answer any TWO of the following:

[2 x 12 =24]

11. a) Explain sample preparation by solid phase microextraction method with a neat labelled diagram.
b) Describe the following open tubular columns i) WCOT ii) SCOT with labelled diagrams.
c) Solute A has a partition coefficient of 3 between toluene and water. Suppose that 100 mL of 0.010 M aqueous solution of A is extracted with toluene. What fraction of A remains in the aqueous phase i) if one extraction with 500 mL is performed,
ii) if five extractions with 100 mL are performed. (4+4+4)

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12. a) Describe suppressed-ion cation chromatography with a neat labelled diagram.
 b) Explain radial paper chromatography.
 c) What is bonded stationary phase in HPLC? Write the chemical reaction involved in the preparation of bonded stationary phase. (6+3+3)
13. a) Explain the working of an evaporative light scattering detector with a labelled diagram.
 b) Calculate the number of theoretical plates obtained for a column of length 17 cm, as the stationary phase particle size varies from 10, 8 and 5 μm . Write a comment on the resolution of the chromatograms obtained in the above cases, assuming that other parameters remain constant for the column.
 c) Explain the principle of affinity chromatography. (4+4+4)

PART-C

Answer any TWO of the following:

[2 x5 =10]

14. A substance C, which is not retained in the stationary phase, exits from the chromatography column 5.0 min after injection. Another substance D, which is retained exits in 20.0 min. The flow rate of the mobile phase is 25 mL/min.
- How long does D spend in the mobile phase?
 - What is the value of capacity factor for D?
 - What is the value of K_D for C?
 - What is mobile phase volume, V_m ?
 - How many theoretical plates are present if the peak for D has $w_{1/2} = 2.0$ min.
15. Predict the order of elution for the following ions from a cation exchange resin
- Mg^{2+} , Ba^{2+} and Ca^{2+} . Give reason for the order of elution.
 - Al^{3+} and Na^+ . Give reason for the order of elution.
 - Name any one suitable ion exchange resin which can be used to carry out the above elutions. (2+2+1)
16. a) A new student uses thin layer chromatography (TLC) for his/her research project. However, the student obtains poor results even after running the TLC experiment multiple times. On careful examination, it was found that the following errors occurred while performing TLC.
- The student forgot to place the lid on the TLC jar.
 - The solvent level in the developing jar is higher than the origin (spotting line) of the TLC plate.
 - The compounds run as a streak than as a spot on TLC.

Explain how each of these errors lead to poor results.

- b) Predict the order of elution of diethyl ether, 1-butanol, 1-butene on a reverse-phase column. Give reason. (3+2)
