

[This question paper contains 5 printed pages.]

Your Roll No.

5143

B

B.Sc. Prog./III

AC-301 : Separation Methods in Analytical Chemistry
(Admissions of 2005 and onwards)

Time : 3 Hours

Maximum Marks : 75

(Write your Roll No. on the top immediately on receipt of this question paper.)

Attempt Five questions in all.

All questions carry equal marks.

1. (a) What is the difference between physical adsorption and chemisorption? Explain. 2
- (b) Define an adsorption isotherm, when does a 'C' adsorption isotherm occur and why is it called a 'C' adsorption isotherm? 4
- (c) How are silica gel and carbon activated? Why are partially deactivated adsorbents preferred over fully activated ones? 4
- (d) 50 mL of 0.60 M acetic acid was equilibrated with 2.5 g of silica gel. The adsorbent is filtered off and a 25 mL aliquot requires 45 mL of 0.10 M NaOH

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to reach a phenolphthalein end point. Calculate the specific adsorption (mg/g) of acetic acid on silica gel. 5

2. (a) What is the difference in the nature of stationary phase and mobile phase in the conventional liquid-liquid partition chromatography and reverse phase liquid-liquid partition chromatography? Name two supports each for normal LLPC and reverse phase LLPC. 6
- (b) Define retention volume. Explain how each of the following variables affects the retention volume of the solute in LLPC? 6
- (i) Solubility of the solute in the stationary phase.
 - (ii) The density of packing of the column assuming the total volume of the packed column is constant.
 - (iii) The temperature at which the column is operated.
- (c) What advantages does HPLC have over conventional LLPC? Enumerate. 3

3. (a) Define R_f value for paper chromatography. Which is the stationary phase in paper chromatography? 3
- (b) Describe the apparatus used for descending paper chromatography with the help of a diagram. 5
- (c) Name the factors that affect the R_f value of a compound in paper chromatography. Describe any two in detail. 5
- (d) Describe the procedure for spotting components for paper chromatography. 2
4. (a) What are the properties of an ideal detector? 5
- (b) Describe an electron capture detector. 5
- (c) Following data was obtained by gas chromatography on a 2.4 m column. The volume of stationary phase is 118.4 mL while the volume of the mobile phase is 276.6 mL.

Compound	Retention time	$W_{1/2}$ (min)
Air	11.40	—
Methylcyclohexane	80.00	4.56
Methylcyclohexene	84.72	4.92
Toluene	107.20	6.24

Calculate :

- (i) Capacity factor for each
 - (ii) Partition coefficient for each
 - (iii) Selectivity factor for methylhexane and methylhexene. 5
5. (a) Describe a method for regenerating an anion exchange column which was originally in the H^+ cycle. 4
- (b) What do you understand by the term ion exchange selectivity? Enumerate any *three* criteria of ion exchange selectivity. 4
- (c) A 10.0 mL sample of water was placed on an ample quantity of a strong cation exchanger, in the hydrogen form. Some deionized water was then passed through the column to flush out any unbound ions. The column effluent, including all washings, was titrated with 0.10 M NaOH; 62.25 mL was required. Calculate the total milliequivalents of cations per litre of water. 6
- (d) If the separation factor for ions A and B $\alpha_A^B > 1$, what does it signify? 1
6. (a) Give the method of extraction of a component of a mixture using solvent extraction when the solute is in solid form. 8
- (b) How can you use gel electrophoresis for determining the molecular weight of a protein? Describe. 7

7. (a) What is the principle used in separation of components of a mixture in gel chromatography? Explain. 4
- (b) Give a method used for coating glass plates with silica slurry in thin layer chromatography. 4
- (c) A gel filtration column is prepared using a Sephadex gel packing which excludes molecules whose molecular weight is larger than 600,000. The volume within the gel particles which is available for accepting the solutes is 250 mL. The void volume is 80.0 mL.
- (i) What is the expected retention volume for a protein of molecular weight 800,000?
- (ii) What is the expected retention volume of a protein of molecular weight 150,000?
- Defend your answer giving reasons. 7