

**UNIVERSITY OF SWAZILAND
MAIN EXAMINATION 2008/09**

TITLE OF PAPER : **SEPARATION METHODS**

COURSE NUMBER : **C511**

TIME : **THREE HOURS**

INSTRUCTIONS : **ANSWER ANY FOUR
QUESTIONS. EACH QUESTION
CARRIES 25 MARKS.**

You are not supposed to open this paper until permission to do so has been granted by the Chief Invigilator.

QUESTION 1

- (a) Give any six separation techniques and for each technique, indicate the property on which the separation technique is based. (6)
- (b) Benzoic acid, C_6H_5COOH , is distributed between benzene and water:
- (i) State the forms in which the acid occurs and the basic equilibrium involved in each phase.
 - (ii) Show the relationship between the distribution ratio, D and the distribution coefficient, K_D for the species involved
 - (iii) What is the effect of increasing the pH of the aqueous phase on D ? (9)
- (c) What is the advantage of multiple extractions with small portions of the same volume of a solvent over a single extraction with the same volume of solvent? Derive an expression to justify the advantage. (10)

QUESTION 2

- (a) Give the form in which each of the following ions can be extracted as ion pairs and an example of a solvent that can be used to extract the ion pair in each case:
- (i) Fe^{3+} from concentrated hydrochloric acid
 - (ii) MnO_4^- from aqueous solution
 - (iii) UO_2^{2+} from aqueous solution (6)
- (b) What are chelating agents and what is the principle involved in their use for the extraction of metals? Give three examples of chelating agents indicating their names, structures and two metal ions that can be extracted by the chelating agent. (10)
- (c) Define chromatography and indicate four classes of chromatographic processes. Explain briefly the principle involved in each class. (9)

QUESTION 3

- (a) Give three advantages of using a gas as the mobile phase in a chromatographic separation. Give two gases commonly employed as mobile phases in GC analyses, and justify their use for this purpose. (5)

- (b) For the GC technique, enumerate the functions and ideal properties of the following:
- (i) The solid support
 - (ii) The stationary phase. (5)
- (c) Give three advantages of the wall coated, open tubular columns over packed columns for GC analyses. Give an account of these advantages and structural designs which enhance the advantages of these columns. (5)
- (d) For the GC – ‘Flame Ionization Detector’ (FID):
- (i) Draw and label its schematic diagram
 - (ii) Discuss its main features, including advantages and disadvantages when compared to the ‘Thermal Conductivity Detector’ (TCD). (10)

QUESTION 4

- (a) Define both retention time and retention volume. Give an expression relating the two. (3)
- (b) Why is it necessary to apply a correction to the adjusted retention volume during GC – analysis, while it is not necessary for LC – methods? (2)
- (c) Give the expressions for the specific retention volume, V_g under ordinary and ideal conditions. Define all the parameters involved. Discuss how V_g , and hence the retention time, t_r are influenced by temperature. (7)
- (d) What is selective factor? How does its value indicate the degree of separation between adjacent peaks? (3)
- (e) A GC column for which the number of theoretical plates, $N = 2.00 \times 10^4$ produces two peaks for two solutes at $t_{r1} = 3.0$ min and $t_{r2} = 3.5$ min respectively.
- (i) Calculate the extrapolated base width of the elution peak for each solute.
 - (ii) Determine the resolution, R .
 - (iii) Estimate the length of the column if $H = 0.25$ cm.
 - (iv) Calculate the selectivity factor, α , if the retention time for an unretained solute is 0.5 min. (10)

QUESTION 5

- (a) Describe the essential materials required for column chromatography? (3)

- (b) What are the precautionary measures which should be taken to achieve good separation in column chromatography? (10)
- (c) What are the differences between thin layer chromatography (tlc) and column chromatography? (3)
- (d) Describe how the tlc plates can be prepared in the laboratory. (3)
- (e) What is the difference between a 'qualitative tlc' and 'preparative tlc' and what are the precautionary measures to be taken to achieve good separation in tlc? (6)

QUESTION 6

- (a) Differentiate between the following in the HPLC technique:
(i) Analytical and guard columns
(ii) Normal phase chromatography and reverse phase chromatography
(iii) Isocratic elution and gradient elution. (6)
- (b) Give four basic requirements for a HPLC pump. (4)
- (c) Describe two of the different kinds of pumps commonly employed in HPLC. What are the advantages and disadvantages of each? (9)
- (d) What are the advantages of supercritical fluid chromatography over GC and HPLC? (6)