

CHUKA



UNIVERSITY

UNIVERSITY EXAMINATIONS

SECOND YEAR EXAMINATION FOR THE AWARD OF DEGREE OF BACHELOR OF SCIENCE (CHEMISTRY/MATHE) AND BACHELOR OF EDUCATION SCIENCE

CHEM 241: CHEMICAL SEPARATION TECHNIQUES

STREAMS: BSC (CHEM/MATHS) & BED (SCI)

TIME: 2 HOURS

DAY/DATE: WEDNESDAY 15/04/2020

8.30 A.M. – 10.30 A.M.

INSTRUCTIONS: Answer question ONE and any other TWO questions

QUESTION ONE (30 MARKS)

- (a) Describe the principles of traditional methods for sample preparation of solid sample given below and comment on each of them
- (i) Solid-liquid extraction [2½ marks]
 - (ii) Soxhlet extraction [2½ marks]
- (b)
- (i) State and compare five gas chromatography inlets in terms of their advantages and disadvantage [7½ marks]
 - (ii) Give four practical problems associated with liquid-liquid extraction [2 marks]
 - (iii) Outline nine advantages of solid phase extraction (SPE) over liquid-liquid extraction (LLE) [4½ marks]
- (c) (i) A solute S, has a K_D between water and chloroform of 5.00. A 50.00 ml sample of a 0.050 M aqueous solution of the solute is extracted with 15.00 ml of chloroform.
- (I) What is the extraction efficiency for this separation? [2 marks]
 - (II) What is the solutes final concentration in each phase? [2½ marks]
 - (III) What volume of chloroform is needed to extract 99.9% of the solute? [1 mark]

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- (IV) Determine the extraction efficiency for two extractions, for three extractions and the number of extractions required to ensure that 99.9% of the solute is extracted [3½ marks]
- (d) An acidic solute, HA, has an acid dissociation constant of 1.0×10^{-5} , and a partition coefficient between water and benzene of 3.00. Calculate the extraction efficiency when 50.0 ml of a 0.025 M aqueous solution of HA buffered to a pH of 3.0 is extracted with 50.0 ml of benzene. Repeat for cases in which the pH of the aqueous solution is buffered. [2 marks]

QUESTION TWO (20 MARKS)

- (a) (i) State two most common types of derivatization [1 mark]
- (ii) Discuss the advantages and drawbacks of the following:
- (I) Precolumn derivatization [3 marks]
- (II) Post-column derivatization [3 marks]
- (b) (i) A hydrodynamic injection is made by applying a pressure difference of 2.5×10^3 pa (approximately 0.02 atm) for 2S to a 75-cm long capillary tube with an internal diameter of $50 \mu\text{m}$. Assuming that the buffer solutions viscosity is $10^{-3} \text{ kg m}^{-1} \text{ s}^{-1}$, what volume of sample is injected? And also calculate the length of the injected sample plug. [3 marks]
- (ii) Explain how HPLC differs from GC in terms of operation, accuracy, precision, sensitivity, selectivity, time, cost and equipment necessary. [2½ marks]
- (iii) In a separation of a mixture of hydrocarbons, the following adjusted retention times were measured
- Propane 2.23 min
Isobutene 5.71 min
Butane 6.67 min
- What is the Kovat's retention index for each of these hydrocarbons? [2 marks]
- (c) (i) In a chromatographic analysis of lemon oil a peak for limonene has a retention time of 8.36 min with a baseline width of 0.96 min. Y- Terpinene elutes at 9.54 min, with a baseline width of 0.64 min. What is the resolution between the two peaks? 1 mark]

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- (ii) In a quantitative analysis of a mixture of three organic compounds by gas chromatography the detector response and the peaks areas respectively of the components were reported as follows: component A – 0.85 and 3.35 cm², components B – 1.0 and 4.2 cm², and component C – 0.95 and 2.8 cm². Calculate the percentage composition of the sample mixture by internal normalization. [3½ marks]
- (iii) Outline four fundamental requirements for a useful resin. [1 mark]

QUESTION THREE (20 MARKS)

- (a) (i) State two advantage of Thin layer chromatography over paper chromatography [2 marks]
- (ii) Outline three methods which can be used to determine quantitatively the amount of the components from the spot of it on the TLC plate [1½ marks]
- (b) (i) Describe the principle of ion exchange chromatography [5½ marks]
- (ii) The ion exchange properties of an ion exchanger are independent of the pH of the mobile phase. Will this phenomenon hold good if the ion-exchanger be carboxylate group in an exchanger or a tertiary amine which can also be used in ion-exchange chromatography? Explain [2½ marks]
- (iii) Explain what happens during the whole process from the beginning when the analyte comes in contact with ion-exchanger to the end when separation is complete by ion exchange [4 marks]
- (c) Discuss the merits and demerits of Uv-visible light sensitive detector used in HPLC [4½ marks]

QUESTION FOUR

- (a) (i) List six advantages of two dimensional thin layer chromatography (TLC) which has contributed for its widely used as a technique of separation [4 marks]
- (ii) List factors that influence resolution in size exclusion chromatography which are related to the following:
- (I) Medium – related factors [1 mark]
- (II) Column – related factors [1 mark]
- (III) Chromatography system – related factors [1 mark]
- (IV) Experimental – related factors [1½ marks]

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(b) A student was performing analysis of protein sample using size exclusion chromatography and the peak of interest was poorly resolved from other major peaks. State the possible causes and the remedy for this [3½ marks]

(c) (i) Electrophoresis is an important group of separation techniques in chemistry and biology. In biology gel electrophoresis is the main type used, while capillary electrophoresis is more popular in chemistry. Give three advantages of capillary electrophoresis over gel electrophoresis and two advantages of gel electrophoresis over capillary electrophoresis [2½ marks]

(ii) Imagine that you need to separate a mixture that contains the components given in the table below. You decide to separate the components by capillary zone electrophoresis in an untreated capillary with the injection done at the positive end of the capillary and the detector is at the negative end. Determine the elution order of the components. Explain why the components come out in this order.

Analyte	1	2	3	4	5	6	7
Charge	+1	-2	0	-1	0	+1	+3
Radius (Å)	1.0	5.0	10.0	3.0	25.0	3.0	2.5

[5½ marks]
