SOUTH EASTERN KENYA UNIVERSITY

UNIVERSITY EXAMINATIONS 2016/2017

SECOND SEMESTER EXAMINATION FOR THE DEGREES OF BACHELOR OF SCIENCE(CHEMISTRY)

SCA 303:SEPARATION AND VOLTAMMETRIC ANALYSIS

DATE: 10TH APRIL, 2017 **TIME: 1.30-3.30**

INSTRUCTIONS TO CANDIDATES

(a) Answer question One and any other Two questions

(b) Question 1 carries 30 marks while the other questions carry 20 marks each

(c) Illustrate your answers with well label diagrams where applicable

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Question 1 (30 marks)

a) Define the following terms as used in chromatography:

i. Chromatogram

ii. Volume flow rate

iii. Retention volume

iv. Capacity factor

(4 marks)

b) Derive the equation for the concentration of a solute [A_n], remaining in an aqueous solution after *n* extractions with an organic solvent. (6 marks)

c) The distribution constant, K, for compound X between n-hexane and water is 8.9. A 50.0 mL solution of 0.200 M of Compound X is to be extracted using a single extraction with 40 mL and five portions of 5.00 mL hexane, respectively. Explain which extraction would be more efficient. (6 marks)

d) State one kind of analyte (compound) to which the following gas chromatography detectors respond to.

i. Thermal conductivity detector

ii. Flame ionization detector

iii. Electron capture detector

iv. Photoionization detector

e) (4 marks)

i. A chromatography column with a length of 10.3 cm and inner diameter of 4.61 mm is packed with stationary phase that occupies 61 % of the volume. If the volume flow rate is 1.13 mL/ min, calculate the linear flow rate in cm/min. (2 marks)

ii. Determine the time it takes for solvent (which is the same as the unretained solute) to pass through the column. (2 marks)

f) Describe the necessary condition under which split, splitless and on-column injection in gas chromatography can be used. (6 marks)

Question 2 (20 marks)

a)

i. Solute S has a partition coefficient of 4.0 between water and chloroform. Calculate the concentration of S in the chloroform if $[S_{(aq)}]$ is 0.020 M. (3 marks)

ii. Given the volume of water is 80.0 mL and the volume of chloroform is 10.0 mL, find the mole ratio of the mixture (i.e., moles of S in $CHCl_3$/ moles of S in H_2O) (3 marks)

iii. The solute S is initially dissolved in 80.0 mL of water. It was then extracted six times with 10.0 mL of $CHCl_3$. Find the fraction of solute remaining in the aqueous phase. (4 marks)

b) State the one advantage for:

i. Temperature programming in gas chromatography

ii. Pressure programming in gas chromatography (4 marks)

c) Explain the following statements:

i. Normal-phase chromatography generally uses non-aqueous solvents and eluent strength increase as the solvent becomes more polar. (2 marks)

ii. Reversed-phase chromatography generally uses aqueous organic solvents and eluent strength increases as the fraction of organic solvent increases (2 marks)

iii. Hydrophilic interaction chromatography uses aqueous organic solvent and the strength increases as a fraction of water in the aqueous solvent increases.

(2 marks)

Question 3 (20 marks)

Describe the order of decisions in method development for gas chromatography.

(20 marks)

Question 4 (20 marks)

a) Define electroosmotic flow (2 marks)

b) Explain clearly how it occurs (4 marks)

c) Describe the principle of separation by capillary zone electrophoresis (7 marks)

d) A certain inorganic cation has an electrophoretic mobility of 5.13×10^{-4} cm^2 s^{-2} V^{-1}. This ion has a diffusion coefficient of 9.1×10^{-6} cm^2 s^{-1}. If this ion is separated by capillary zone electrophoresis with a 50 cm capillary:

i) Calculate the expected plate count, N, at applied voltage of 20 kV (2 marks)

ii) Under the separation conditions, the electroosmotic flow rate was 0.65 mm s^{-1} towards the cathode. If the detector was placed 40 cm from the injection end of the capillary, determine the time it takes, in minutes, for the analyte to reach the detector after the applied field. (5 marks)

Question 5 (20 marks)

a) State **Four** properties that a resin should poses for efficient separation. (4 marks)

b) Explain **Five** applications of ion exchange chromatography (5 marks)

c) Calculate the minimum distribution coefficient that permits removal of 99 % of solute from 50.0 mL of water with two 25.0 mL extraction with toluene. (6 marks)

d) List the variables that lead to band broadening in chromatography. (5 marks)