SOUTH EASTERN KENYA UNIVERSITY

UNIVERSITY EXAMINATIONS 2016/2017

FIRST SEMESTER EXAMINATION FOR THE DEGREE OF BACHELOR OF SCIENCE(CHEMISTRY)

SCA 303: SEPARATION AND VOLTAMMETRIC ANALYSIS

DATE:16 TH DECEMBER, 2016 TIME:10.30-12.30 P.M

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

Question 1 (30 marks)

a) Describe the following terms as used in separation:

i) Elution

ii) Stationary phase

iii) Retention time

iv) Selectivity factor **(6 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 packed column in a Gas chromatography had an inside diameter of 5.0 mm, the measured volumetric flow rate at the column outlet was 48.0 mL/min. If the column porosity was 0.43, calculate the linear velocity in cm/s **(3 marks)**

d.) Describe the physical difference between capillary and packed columns. State the

advantage and disadvantage of each. **(5 marks)**

e)List **Three** advantages of fused silica capillary columns compared with glass or metal columns (**3marks)**

f.) Explain how the instruments in supercritical fluid chromatography differ from those of GC and HPLC. **(4 marks)**

g.) In a polarographic experiment, a limiting current was applied for 45 minutes in a 60mL of 0.08 M Cu^{+2}. If the average current during the time of experiment was 6.0 µA , Calculate the fraction of copper removed from the solution. **(3 marks)**

Question 2 (20 marks)

a) Describe the principle on which each of the following GC detectors are based:

i) Thermal detector **(1.5 marks)**

ii) Flame ionization **(1.5 marks)**

iii) Electron capture **(1.5 marks)**

iv) Photoionization detector **(1.5 marks)**

b) State **Two** advantages and **One** disadvantage of each of the detectors in 2(a) above. **(6 marks)**

c) List the type of substances to which each of the following chromatographic method is most applicable:

i) Gas-liquid
ii) Ion
iii) Gel permeation
iv) Chiral **(4 marks)**

d) Indicate the order of elution of the following compounds: *benzene, diethylether and n-hexane* from a reverse-phase HPLC column and *ethyl acetate, acetic acid and dimethylamine* from a normal-Phase HPLC column, respectively.

(4 marks)

Question 3 (20 marks)

a) State the properties of supercritical that are important in chromatography.

(2 marks)

b) The following data are for a liquid chromatographic column:

Length of Packaging	24.7
Flow rate	0.313 mL/ min
Vm	1.37 mL
Vs	0.164 mL

A chromatogram of a mixture of A, B, C and D provided the following data:

	Retention Time, min	Width of Peak Base (W), min
Nonretained	3.1	-
A	5.4	0.41
B	13.3	1.07
C	14.1	1.16
D	21.6	1.72

Calculate, for A, B, C and D:

i) The number of theoretical plates

ii) The retention factor

iii) The distribution constant

(8 marks)

c) List the variables that lead, in gas-liquid chromatography, to:

i) Band broadening
ii) Band separation

(4 marks)

d) Explain:

i) Bonded and cross-linkedchromatographic stationary phase **(2 marks)**

ii) Why are chromatographic stationary phase bonded and cross-linked

(2marks)

Question 4 (20 marks)

a) Distinguish between:

i) Voltametry and amperometry

ii) Differential-pulse voltammetry and square-wave voltammetry

iii) A limiting current and diffusion current:

iv) The standard electrode potential and half-wave potential for a reversible reaction at a working electrode.**(10 marks)**

b) Explain why a high supporting electrolyte concentration is used in most analytical procedures. **(4 marks)**

c) State the reason for buffering solutions in organic voltammetry **(2 marks)**

d) Describe the purpose of electrodeposition in stripping analysis **(2 marks)**

e) The equation below is for current/ voltage relationship for a reversible reaction,

$$E_{appl} = E_{1/2} - \frac{0.0592}{n} \log \frac{i}{i_1 - i}$$

Explain how it is used to determine the number of electrons, n, involved in a reversible reaction at an electrode. **(2 marks)**

Question 5 (20 marks)

a) State 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)**