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(University of Choice)

MASINDE MULIRO UNIVERSITY OF SCIENCE AND TECHNOLOGY (MMUST)

MAIN CAMPUS

SPECIAL/SUPPLEMENTARY EXAMINATIONS

2021/2022 ACADEMIC YEAR

SECOND YEAR FIRST SEMESTER EXAMINATIONS

FOR THE DEGREE

OF

BSc (CHEMISTRY) AND BSc (INDUSTRIAL CHEMISTRY)

COURSE CODE: **SCH 220**

COURSE TITLE: **ANALYTICAL CHEMISTRY II**

DATE: *Tuesday 26/07/2022* **TIME:** *8.00 – 10.00 AM*

INSTRUCTIONS TO CANDIDATES

Answer all the Questions

TIME: 2 Hours

MMUST observes ZERO tolerance to examination cheating

This Paper Consists of 4 Printed Pages. Please Turn Over.

QUESTION ONE [21 Marks]

a) State two factors that influence the rate of evaporation. [2 Marks]

b) Define the following terms: [2 Marks]

(i) Supersaturated solution

(ii) Polymorphism

c) Crystallization proceeds via nucleation. Primary nucleation can be modeled by the following equation.

$$B = \frac{\partial N}{\partial t} = k_n(C - C^*)$$

From the equation above, define the following terms

(i) B

(ii) N

(iii) K_n

(iv) C

(v) C*

(vi) (C – C*) [6 Marks]

d) Liquid-liquid extraction is one of the most widely used methods in sample preparation.

(i) What do you understand by the term 'liquid-liquid extraction' [1 Mark]

(ii) 15 mL of an aqueous 0.15M solution of butyric acid is shaken with 10 mL of chloromethane. After the layers are separated, it was determined by titration that 0.75 millimole of butyric acid remained in the aqueous layer. Calculate:

(I) The distribution ratio of butyric acid in the two phases [2 Marks]

(II) The percentage of butyric acid extracted from the aqueous phase [2 Marks]

e) Aqueous two-phase extraction (ATPE) is a unique form of solvent extraction.

(i) State any two types of aqueous two-phase extractions [2 marks]

(ii) State three advantages ATPE over traditional solvent extraction technique [3 marks]

QUESTION TWO [15 Marks]

a) Briefly explain the difference between;

(i) Adsorption chromatography and partition chromatography [2 Marks]

(ii) Normal phase chromatography and reverse phase chromatography [2 Marks]

b) An analytical chemistry student has a mixture of cyclohexane (BP = 81^0C and toluene (BP = 111^0C) in the ratio 1:1. She requires cyclohexane of purity above 95%. She decides to use simple distillation method.

(i) Explain how she would achieve this. [3 Marks]

(ii) What would be the advantages of her using fractional distillation method in place of simple distillation? [2 Marks]

c) A chromatogram obtained from analysis of a sample using HPLC instrument gave the solvent peak after 8 minutes. The peak for a component A in the sample appeared after 18.6 minutes. If the bandwidth of the peak of A is 1.2 minutes, calculate the:

(i) The number of theoretical plates in the column used. [2 Marks]

(ii) The effective plate number of the column. [2 Marks]

(iii)The retention factor for the component A in the column [2 Marks]

QUESTION THREE [18 Marks]

a) Define the following terms: [2 Marks]

(i) Filtration

(ii) Relative volatility

b) The van Deemter equation, below, explains the sources of peak broadening in a packed chromatography column:

$$H = A + \frac{B}{u} + C\bar{u}$$

Briefly explain the meaning of each of the following terms in the van Deemter equation:

(i) H

(ii) $\bar{u}$

(iii) A

(iv) B

(v) C

c) Ethanol and methanol were separated in a capillary column with the retention times of 295 seconds and 315 seconds respectively on a 30 cm column. An unretained air peak occurred at 15 seconds. Calculate:

(i) The separation factor between the two peaks [2 Marks]

(ii) The resolution between the two peaks in the column used. [5 Marks]

(iii)Determine the length of the column required to give a resolution of 1.75 [4 Marks]

QUESTION FOUR [16 Marks]

a) An iron ore analyzed by dissolving 1.1324 g sample in concentrated hydrochloric acid. The resulting solution was diluted with water and the iron(III) Was precipitated as a hydrous oxide $Fe_2O_3.x\ H_2O$ by addition of NH_3. After filtration and washing, the residue was ignited at a high temperature to give 0.5394g of pure Fe_2O_3. (Fe = 55.87g/mol, O = 16.00g/mol).

(i) Calculate the mass of iron in the sample [4 Marks]

(ii) Determine the percentage of iron in the sample. [2 Marks]

b) The action of an alkaline I_2 solution on the rodenticide warfarin, $C_{19}H_{16}O_4$ (308.34g/mol), results in the formation of 1 mol of iodoform (CHI_3) for each mole of the parent compound reacted. Analysis for warfarin can then be based on the reaction between CHI3 and Ag+.

$$CHI_3 + 3Ag^+ + H_2O \longrightarrow 3AgI(s) + 3H^+ + CO(g)$$

The CHI_3 produced from a 13.96g sample was treated with 25.00 mL of 0.02979 M $AgNO_3$, and the excess Ag+ was then titrated with 2.85 mL of 0.05411 M KSCN. Calculate:

(i) The moles of Ag+ that reacted with KSCN [2 Marks]

(ii) The moles of CHI_3 which reacted with $AgNO_3$ [3 Marks]

(iii)The percentage of warfarin in the sample used [5 Marks]