

**KIBABII UNIVERSITY**  
**UNIVERSITY EXAMINATIONS**  
**2021/2022 ACADEMIC YEAR**

**SECOND YEAR SECOND SEMISTER EXAMINATION**  
**FOR THE DEGREE**  
**OF**  
**BACHELOR OF EDUCATION (SCIENCE)**

**COURSE CODE:** SCH 229

**COURSE TITLE:** CHEMICAL ANALYSIS AND STRUCTURE DETERMINATION

**INSTRUCTION:** Answer question one and any other two questions. Start an answer on a new page in the answer booklet

**DATE:** 16/05/2022

**TIME:** 9:00AM-11:00AM

This paper contains 6 printed pages

**Question one (30 marks)**

a i) The table below represents PXRD data

Compound name	Chemical formula	Mineral percentage
Illite	$\text{Al}_4\text{K Si}_2\text{O}_{12}$	55.1
Petalite	$\text{AlLiSi}_4\text{O}_{10}$	5.9
Ulvospinel	$\text{Fe}_{2.247}\text{Ti}_{0.751}\text{O}_4$	4.9
Kaolinite	$\text{Al}_2\text{H}_4\text{Si}_2\text{O}_9$	5.3

Use the table above to discuss some of the applications of PXRD

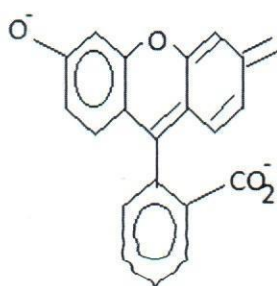
**(3 marks)**

b) Sketch and explain the instrumentation flow diagram for non-destructive Fourier Transform infra-red spectroscopy

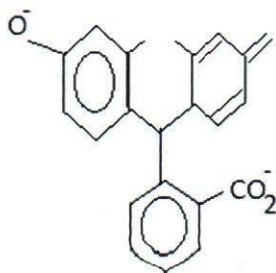
**(4 marks)**

c) Explain which of the molecules A and B fluorescence

**(3 marks)**



A



B

d) Identify steps in chemical analysis of a soil sample to identify presence of iron III **(3 marks)**

e) How would variation in magnetic field affect an NMR analysis of an analyte from the bark of a tree **(2 marks)**

e) Calculate fraction of chelate extracted when 25 ml of  $4.3 \times 10^{-2}$  M of the metal chelate shaken with 2 successive 5ml portions of chloroform. The distribution coefficient is 4.4 **(3 marks)**

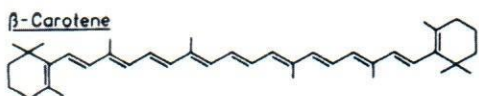
f) Four compounds were separated on a chromatographic column whose length was 24.7 cm. The following results were obtained.

Compound	Retention time/min	Width of peak/min
A	14.1	0.41
B	13.3	1.07
C	5.4	1.16
D	21.6	1.72

- i) Sketch the chromatogram with a poor resolution for the second and third peaks, assuming increasing concentration as affinity for mobile phase increases **(2 marks)**
- ii) Calculate the plate height for the first compound **(2 marks)**
- g) i) Briefly explain why HCl is opaque to IR radiation **(2 marks)**
- ii) Briefly explain what FTIR is **(3 marks)**
- iii) What is anisotropy **(2 marks)**

**Question 2 (20 marks)**

- a) Discuss the difference between Photophorescence and Photoluminiscence as used in UV analysis giving examples **(4 marks)**
- b) The UV spectrum of propanone shows 3 peaks at 280, 188, and 154 nm.
- i) What chromophore is responsible for each transition **(3 mark)**
- ii) Explain the difference in the  $\lambda_{\max}$  **(3 marks)**
- c) Identify 2 challenges experienced when using UV spectrophotometer for analysis **(2 marks)**
- d) Beta carotene gives carrot its red colour due to conjugation of 10 bonds. How would you conver this yellow into blue **(2 marks)**

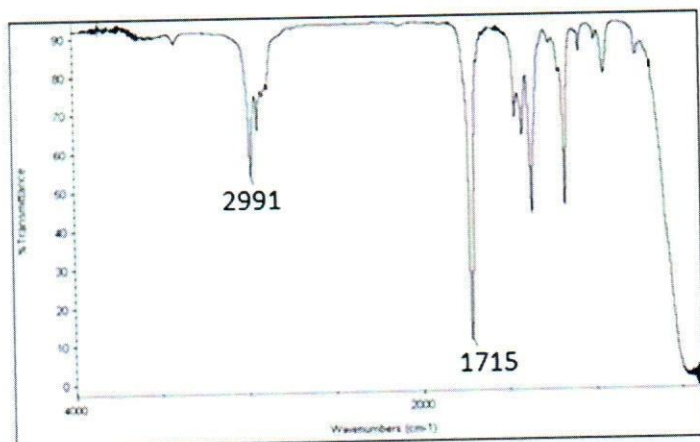


- e) Use woodward fischer rules to calculate labda maxima for the molecule below **(2 marks)**

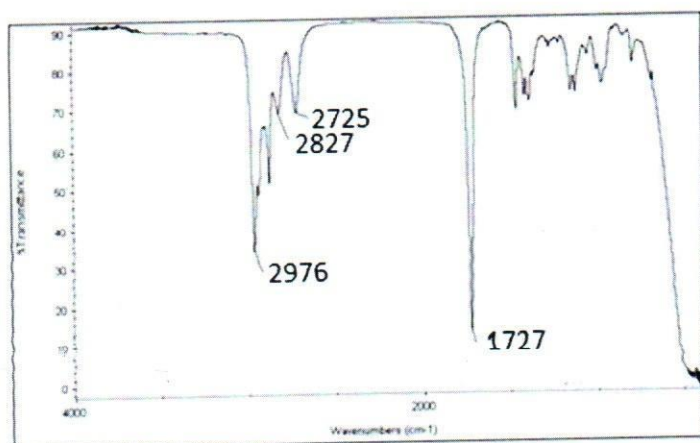


- f) Study the spectra of A and B below for a compound with the molecular formula 72

Spectrum of A



Spectrum of B



- i) assign functional groups to the bands labeled in A
- ii) Predict the structural formula of A and B

(2 marks)

(2

marks)

**Question 3 (20 marks)**

- a) Briefly explain the mode of separation in paper chromatography (2 marks)
- b) Show that the distribution coefficient of acetic acid is not equal to its distribution ratio (2 marks)
- c) i) Explain 3 factors that enhance molecular fluorescence (6 marks)
- ii) State 2 factors that may cause non-resonance fluorescence (4 marks)
- d) Briefly state 2 differences between EDX and MF (2 marks)
- e) Sketch the NMR spectra of  $\text{CH}_3\text{CH}_2\text{OH}$  (4 marks)

**Question 4 (20 marks)**

a) Calculate the frequency at which a proton nucleus would absorb in a M.F of 25.14 ( $h = 6.6 \times 10^{-34} \text{ m}^2 \text{ kgs}^{-1}$ ,  $B_n = 5.5 \times 10^{-27}$ , and  $g_i$  for  $^{13}\text{C} = 1.404$ ) (2 marks)

b) The following NMR absorptions of 2.1  $\delta$ , given in  $\delta$  units, were obtained on a spectrometer operating at 300 MHz. Convert the chemical shifts from  $\delta$  into hertz downfield from TMS.

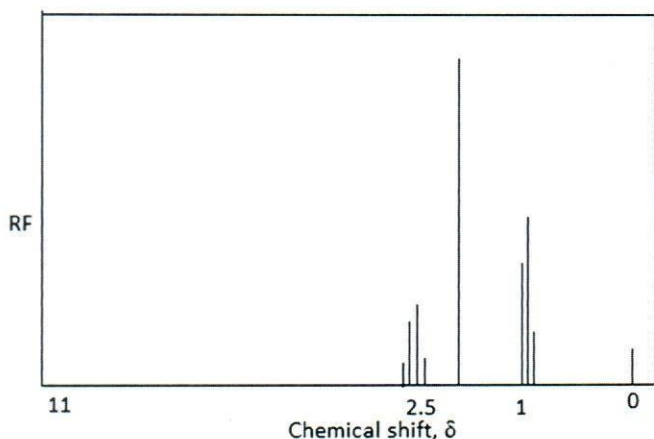
a) on 300 MHz;

c) What is meant by

i) chemical shift in NMR (2 marks)

ii) spin-spin splitting (2 marks)

d) The compound whose  $^1\text{H}$  NMR spectrum is shown below has a formula  $\text{C}_4\text{H}_8\text{O}$  and has an IR absorption peak at  $1740 \text{ cm}^{-1}$ . Propose its structure. (4 marks)



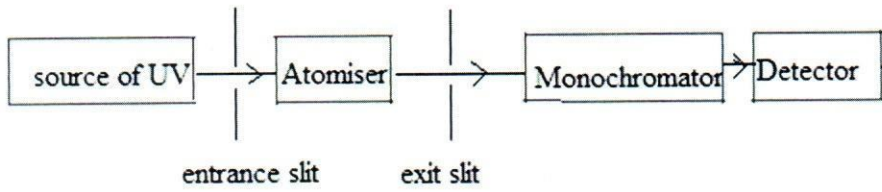
e) 10 ml of water containing copper was treated with a reagent which formed a deep blue colored complex containing copper. The resulting solution diluted to 100ml using deionized water. This solution gave an absorbance of 0.357 at 460 nm. To another 10 ml of the water sample 5ml of 0.2mg/ml copper solution was added and the solution made to 100ml using deionized water. This solution gave an absorbance of 0.42. Calculate the concentration of copper in water in mg/ml

(5 marks)

f) Explain the difference between the red and blue shift in UV spectroscopy (5 marks)

**Question 5(20 marks)**

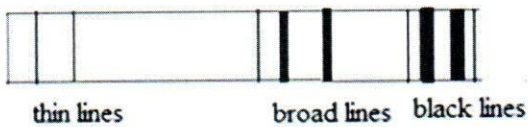
a) i) Discuss the major difference as observed on the instrumentation schemes below (4 marks)



and

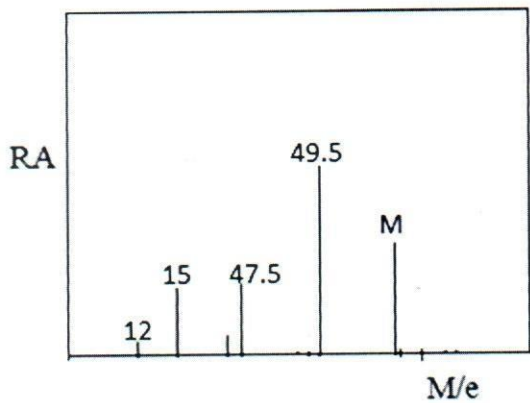


ii) Explain why some lines are broader than others on the AAS spectrum below; (4 marks)



b) Discuss three modes of ionization for the mass spectrometer (6 marks)

c) Study the mass spectrum of a molecule below isolated from an analysis.



i) Assign possible fragments for 15, 47.5 and 49.5 (4marks)

(4marks)

ii) Explain the significance of M (2 marks)

(2 marks)