MASINDE MULIRO UNIVERSITY OF SCIENCE AND TECHNOLOGY (MMUST)

Main campus

UNIVERSITY regular EXAMINATIONS

2022/2023 ACADEMIC YEAR

FOURTH YEAR SECOND SEMESTER EXAMINATIONS

FOR THE DEGREE

OF

Bachelor of Science (CHEMISTRY, INDUSTRIAL CHEMISTRY)

COURSE CODE: SCH 221

COURSE TITLE: INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

DATE: 25/04/2023 **TIME:** 12.00-2.00 PM

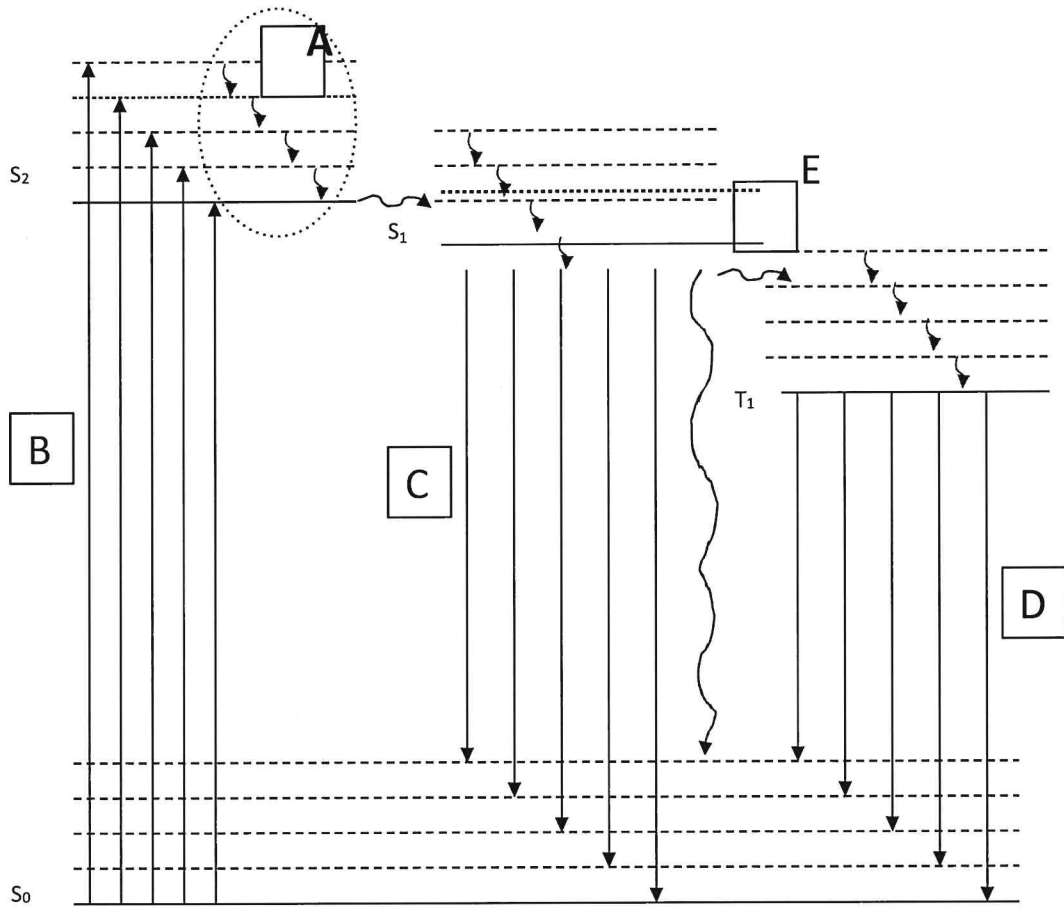INSTRUCTIONS TO CANDIDATES

Attempt all questions

TIME: 2 Hours

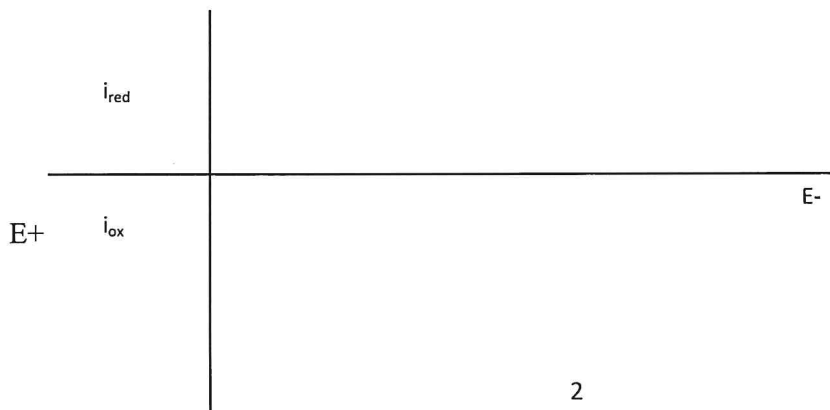MMUST observes ZERO tolerance to examination cheating

This Paper Consists of 5 Printed Pages. Please Turn Over.

Question 1

(a) Label the transitions (the large letters) in the following diagram (5 marks)

S_2

A

E

S_1

B

C

T_1

D

S_0

(b) Sketch a cyclic voltammogram of a hypothetical reversible redox-couple: $A + e = A^-$ (5 marks)

i_{red}

E-

E+ i_{ox}

2

(c) A differential pulse polarogram gave a peak current of 10.1 μA for Cd^{2+} reduction for a 20.00 ml water sample. A spike of 0.0100 ml of 0.025 M of Cd^{2+} to this sample volume gives a current of 23.1 μA. What is the concentration of Cd^{2+} in the original sample?

Hint: consider that the volume after that spike did not significantly change (20.00 vs 20.01 ml) (7 marks)

Question 2

(a)List four types of motions executed by vibrating I.R .molecules.(4 marks)
(b) A fundamental I.R absorption peak has a frequency μ =1051 cm^{-1} Calculate the frequency of first overtone.(2marks)

(c) Describe the infrared spectroscopic method of determination of concentration of unknown sample. (6 marks marks)
(d) The presence of an impurity X in propane was determined using absorption band of X standard at $2041 cm^{-1}$ The data obtained is given below;

%X	1.00	2.00	3.00	4.00
Absorbance	0.240	0.481	0.719	0.915

(i)define the figure 2041 cm^{-1} stating what it reflects in terms of FTIR (2 marks)
(ii) calculate the percentage of X in an unknown sample that had an absorbance of 0.624 using the same cell and same instrument.(4marks)

Question 3

(a) Describe and give three main chemical interferences seen in atomic absorption spectroscopy (6 marks)

(b) An ore is suspected to contain Zinc .Describe the AAS analytical procedure for;
(i) Confirmation of presence of zinc in the ore. (6 marks)
(ii) Determination of concentration level of zinc in the ore.(6 marks)

Question 4

   (a) Define the following terms as used in NMR spectroscopy (4 marks)
      (i)   Shielding
      (ii)   De-shielding
   (b) Using appropriate diagrams explain why increasing the alignment of the magnetic moment of an atomic with the applied magnetic field strength results in an increased signal in NMR. (5 marks)
   (c) The ^1H NMR spectra of N-sec-butylaniline obtained on 60 MHz and 220 MHz instruments is shown below:
    (i) Discuss the effect of the magnetic field strength in NMR spectral analysis (2 marks)
      (ii)   Based on the chemical shifts shown, which protons are most heavily shielded? Explain. (2 marks)
      (iii)   Why are the resonances for the _6_ and _7_ protons split into 2 and 3 peaks, respectively? (2 marks)
      (iv)   Discuss the difference in spectra between position 1 and 3 (2 marks)

(a) 60 MHz

(b) 220 MHz