



MACHAKOS UNIVERSITY

University Examinations 2019/2020 academic Year

SCHOOL OF PURE AND APPLIED SCIENCES

DEPARTMENT OF PHYSICAL SCIENCES

FOURTH YEAR SPECIAL/SUPPLEMENTARY EXAMINATION FOR

BACHELOR OF EDUCATION (SCIENCE)

SCH 404: INSTRUMENTAL METHODS

DATE: 22/1/2021

TIME: 2.00-4.00 PM

INSTRUCTIONS:

- The paper consists of **two** sections.
- Section **A** is **compulsory**.
- Answer any **two** questions from section **B**.

Required Data

$$h = 6.6262 \times 10^{-34} \text{ Js}$$

$$c = 2.998 \times 10^8 \text{ ms}^{-1}$$

$$0^\circ\text{C} = 273.15\text{K}$$

SECTION A - COMPULSORY. [30 MARKS]

QUESTION ONE (30 MARKS)

- a) State the FOUR main components of optical molecular spectrophotometry. (2 marks)
- b) Beryllium (II) forms a complex with acetylacetone (166.5 g/mol). Calculate the molar absorptivity of the complex, given that a 2.25 ppm solution has a transmittance of 37.5 % when measured in 1.00 cm cell at 295 nm, the wavelength of maximum absorbance. (3 marks)
- c) State three radiation sources and three detectors used in Infrared spectroscopy (3 marks)
- d) Describe the principle on which each of the following GC detectors is 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)
- e) **List and explain** three factors that cause spectral lines to broaden (6 marks)
- f) State Four ways used to introduce solid samples in the atomizer of atomic absorption/emission spectrometers (4 marks)
- g) State **Two** advantages and **One** disadvantage of each of the detectors in 1(d) above. (6 marks)

QUESTION TWO (20 MARKS)

- a) State the relationship between:
 - i) Absorbance and transmittance
 - ii) Absorptivity and molar absorptivity (2 marks)
- b) i Express an absorbance of 0.0356 in terms of percent transmittance. (2 marks)
- ii A solution containing the complex formed between Bi(III) and Thiourea has a molar absorptivity of $9.32 \times 10^3 \text{ Lcm}^{-1}\text{mol}^{-1}$ at 470 nm. Calculate the absorbance of a $5.67 \times 10^{-5} \text{ M}$ solution of the complex in a 1.00 cm cell. (3 marks)
- c) It has been discovered that the water in the Marumba reservoir has been contaminated with a water soluble paint discharged at the intake point. A sample of the water has been presented to the chemistry laboratory for determination of the concentration of the suspected paint. Describe a method development procedure to be used for the analysis of the sample using UV-Vis spectrophotometer (9 marks)
- d) Explain the main difference between:
 - i) Fourier Transform Infrared spectrophotometer (FT) and Dispersive infrared spectrometer (DIR) (2 marks)
 - ii) The position of the sample compartment in IR and in UV-Vis spectrometers (2 marks)

QUESTION THREE (20 MARKS)

- a) Describe the basic differences among atomic absorption, atomic emission and atomic fluorescence spectroscopy (6 marks)
- b) Explain FOUR characteristics that an ideal atomization-excitation source should poses. (8 marks)
- c) State the THREE sources of non-spectral interference and explain how each can be eliminated citing examples were possible (6 marks)

QUESTION FOURTH (20 MARKS)

- a) Distinguish between:
- Voltammetry and amperometry
 - Differential-pulse voltammetry and square-wave voltammetry
 - A limiting current and diffusion current:
 - 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,

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

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

QUESTION FIVE (20 MARKS)

The molar absorptivities data for the cobalt and nickel complexes with 2,3-quinoxalinedithiol are as shown in the table below

$\lambda_{\max}$	Molar absorptivity, ϵ	
	Co	Ni
510	36400	5520
656	1240	17500

A 0.425 g sample was dissolved and diluted to 50 mL. A 25.0 mL aliquot was treated to eliminate interference; after addition of 2,3-quinoxalinedithiol, the volume was adjusted to 50.0 mL. This solution had an absorbance of 0.446 at 510 nm and 0.326 at 656 nm in a 1.00 cm cell.

- a) Determine the concentration of Cobalt and Nickel in mol/Litre (14 marks)
- b) Calculate the concentration (in $\mu\text{g/g}$) of cobalt and nickel in the sample. (6 marks)