# MACHAKOS UNIVERSITY

**University Examinations 2018/2019**

**SCHOOL OF PURE AND APPLIED SCIENCES**

**DEPARTMENT OF PHYSICAL SCIENCES**

**FOURTH SUPPLEMENTARY EXAMINATION FOR THE BACHELOR OF EDUCATION (SCIENCE) AND BACHELOR OF EDUCATION (SPECIAL NEEDS)**

**SCH 404: INSTRUMENTAL METHODS**

**DATE: 24/7/2019** **TIME: 11:00 – 1:00 PM**

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**INSTRUCTIONS:**

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

**Required Data**

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

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

$0^{o}C = 273.15K$

**SECTION A - COMPULSORY. [30 MARKS]**

**QUESTION ONE (30 MARKS)**

a) State the FOUR main components of optical molecular spectrophotometry.

(2 marks)

b) Berrylium (II) forms a complex with acetyacetone (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 $9.32 \times 10^3$ $Lcm^{-1}mol^{-1}$ at 470 nm. Calculate the absorbance of a $5.67 \times 10^{-5}$ 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 (20MARKS)**

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 FOUR (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 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 µg/g)** of cobalt and nickel in the sample. (6 marks)