UNIVERSITY OF ESWATINI

**Final Examination– 2020/2021**

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| **TITLE OF PAPER:** | **Spectro Chemical Analysis** |
| **COURSE NUMBER:** | **CHE607** |
| **TIME ALLOWED:** | **Three Hours** |

INSTRUCTIONS:

**Answer any four (4) questions of the six (6) questions and every question holds 25 marks. NB: all questions are to be answered in a separate answer sheet. Marks will be deducted for improper units and lack of procedural steps in calculations. Diagrams show be clear, large and properly labelled.**

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This Examination Paper Contains **SIX** Printed Pages Including This Page

***You are not supposed to open the paper until permission to do so has been granted by the Chief Invigilator.***

QUESTION 1

(a) Electromagnetic radiation interacts with matter causing it to undergo different processes depending on how the radiation has affected it.

With the aid of diagrams, describe how each of the following occur when radiation interacts with the matter.

(i) Absorption (3)

(ii) Emission (3)

(iii) Fluorescence (3)

(iv) Phosphorescence (3)

(b) Electromagnetic radiation can be described as waves or as particles called photons.

What are the three (3) physical properties of electromagnetic waves? (3)

(c) By how many KJ/mol is the energy of oxygen increased when it absorbs UV radiation with the wavelength of 147 nm? (5)

*Speed of light* = $2.998 \times 10^{8}\ ms^{-1}$; *Planck's constant* = $6.626 \times 10^{-34}\ Js$

(d) How much is the energy of $CO_2$ increased when it absorbs IR radiation with a +-

(e) wavenumber of 2300 $cm^{-1}$. (5)

QUESTION 2

(a) Why is it preferred to express the Beer-Lambert law using absorbance rather than % transmittance (%T)? (6)

You may use the following data for your explanation.

| Path length | 0 | 0.2 | 0.4 | 0.6 | 0.8 | 1.0 |
|---|---|---|---|---|---|---|
| % Transmittance | 100 | 50 | 25 | 12.5 | 6.25 | 3.125 |
| Absorbance | 0 | 0.3 | 0.6 | 0.9 | 1.2 | 1.5 |

(b) Give three (3) advantages of each of the following:

(i) AC spark over DC spark (3)

(ii) Using a photographic plate as a detector over a photomultiplier tube (3)

(c) Write the three (3) conditions that make the ICP (plasma) eliminate most of the interferences that are experienced with flames. (3)

(d) In atomic spectroscopy, the Doppler Effect and pressure broadening are similar in magnitude and yield the same linewidths of $10^{-3}$ to $10^{-2}$ nm.

Explain how each of the two (2) mechanisms broaden the linewidth.

(i) Doppler Effect (3)

(ii) Pressure broadening (3)

(e) The ICP-OES can experience several interferences during elemental analysis.

Describe each of the following interferences and briefly explain how each can be remediated during the analysis.

(i) Chemical interference (2)

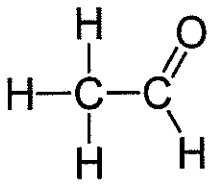
(ii) Ionization interference (2)

QUESTION 3

(a) Define chromophores (2)

(b) Organic molecules undergo different electronic transitions when struck by electromagnetic radiation. The type of transition is determined by the type of bonding in the structure of the molecule.

(i) List all the six (6) electronic transitions exhibited by acetaldehyde,



(6)

(ii) Of the transitions listed in (i), which ones are not possible (forbidden) (2)

(iii) Use an energy diagram to arrange the allowed electronic transitions in order of energy requirement. Justify your order. (6)

(iv) By aid of a diagram, describe how the $\pi \rightarrow \pi^*$ transition occurs (5)

(c) In spectroscopy there are two types of instruments that are used, single and double beam spectrometers.

(i) What is the difference between a single beam and a double beam spectrometer (2)

(ii) Explain how does a double beam instrument remove the background (2)

QUESTION 4

(a) Atomic absorption make use of the hollow cathode lamp to identify and quantify metals in samples.

(i) Draw and label the hollow cathode lamp (3)

(ii) Explain how the hollow cathode lamp works (3)

(b) In classical dissolution the solid sample is acid digested as pretreatment for analysis. The acids are added into the solid sample in the order: nitric acid, sulphuric acid, perchloric acid and lastly hydrofluoric acid then heated in an open vessel.

In this method of digesting samples, explain the role of

(i) nitric acid (2)

(ii) sulphuric acid (2)

(iii) perchloric acid (2)

(iv) hydrofluoric acid (2)

(c) Give **two** (2) precautions that should be taken when carrying out acid digestion (2)

(d) Write **two (2)** limitations of acid digestion (2)

(e) Microwave digestion is another method that is used to digest solid samples for spectroscopic analysis.

(i) Name the sample holder during microwave digestion (1)

(ii) Explain how microwave digestion takes place (3)

(iii) Give one limitation of microwave digestion (1)

(f) Explain the chemical part in ultrasonic dissolution? (2)

QUESTION 5

(a) The ICP is the most preferred method of elemental analysis lately.

(i) Give five (5) advantages of ICP over the AAS in spectroscopy (5)

(ii) List two (2) disadvantages of ICP over FAAS and GFAAS (2)

(b) A typical monochromator using a grating is a rectangular block of glass with 1180 lines etched on every mm of its surface. It is 4.6 cm wide.

(i) Sketch a diffracting grating monochromator showing the incident beam and the monochromatic beam (5)

(ii) State the Bragg's equation for the grating acting as a monochromator, and calculate the primary angle at which radiation of 300 mm is diffracted when it hits the grating. (3)

(iii) Calculate the first order resolving power of this grating, and calculate the resolution at 750 mm (3)

(c) Prisms are widely used in uv-visible spectrometers as monochromators. The base length of a prism is typically 5cm, and the prism material has a dispersion of $2.7 \times 10^{-5}$

(i) State Snell's law for a prism (2)

(ii) Calculate the resolution power of the prism, and the resolution at 5268 A. (3)

(iii) Use diagrams to explain the principle of "Resolution As Limited by the Exit Slit" (2)

QUESTION 6

(a) Matrix effects are problematic in atomic spectroscopy. For each of the following spectroscopic techniques, discuss how matrix effects arise, and state how they can be eliminated in each case.

(i) ICP-OES (3)

(ii) DC Spark (3)

(iii) Flame Atomic Absorption Spectrometry (3)

(iv) Electrothermal Vaporization Atomic Absorption Spectrometry (3)

(b) The Inductively Coupled Plasma (ICP) Optical Emission is now the widely preferred atomic spectroscopic technique.

(i) Give a brief description of the ICP as a source of emission signals (3)

(ii) Use a diagram to show why it is possible for the ICP to measure up to 35 elements simultaneously (3)

(iii) How does the "order of magnitude" of the ICP compare with that of the atomic absorption techniques, and what are the implications of this? (3)

(c) Define the detection limit of an instrument (2)

(d) Describe the 'dissolution problem' in analytical Chemistry in so far as it relates to productivity in atomic absorption spectrometry (2)