MACHAKOS UNIVERSITY

University Examinations for 2021/2022 Academic Year

SCHOOL OF PURE AND APPLIED SCIENCES

DEPARTMENT OF PHYSICAL SCIENCES

FOURTH YEAR FIRST SEMESTER EXAMINATION FOR

BACHELOR OF SCIENCE IN ANALYTICAL CHEMISTRY

SAN 401: SPECTROMETRIC METHODS OF ANALYSIS

DATE: 25/8/2022 **TIME: 2.00-4.00 PM**

INSTRUCTIONS:

- The paper consists of **two** sections.
- Section **A** is **compulsory** (30 marks).
- Answer any **two** questions from section **B** (each 20 marks).

QUESTION ONE (30 MARKS) (COMPULSORY)

a) Electromagnetic radiations consist of electrical and magnetic waves oscillating at right angles to each other. They travel away from the source with the velocity of light. The waves are characterized by frequency, wavelengths and wave number. Define the three terms which characterize magnetic waves and indicate the symbols used to represent each. (3 marks).

b) The analytical process is the science of taking measurements in an analytical and logical way. It involves identifying or quantifying an analyte in a complex sample. Clearly name and explain the main steps involved in an analytical process. (7 marks)

c) An infrared (IR) spectrometer subjects a compound into infrared radiation in the 5000 – 667 cm^{-1} range. This radiation does supply sufficient energy for bonds in the molecule to vibrate by stretching or bending. As the molecule is subjected to the individual wavelengths in the 5000 – 667 cm^{-1} range, it absorbs only those possessing exactly the energy required to cause a particular vibration. Name and explain the two sections usually studied in an infrared spectrum. (4 marks)

d) High performance liquid chromatography is a separation technique used by analysts to undertake qualitative and quantitative analysis. There are a number of modes of HPLC available to the analyst. Differentiate between the two modes of HPLC, reversed phase chromatography and normal phase chromatography. (6 marks)

e) In mass spectroscopy, the ion source is where the sample of interest is ionized with a positive or negative charge and converted into the gas phase. Briefly explain the working of Electron Impact and Chemical ionization as applied in Mass Spectroscopy. (4 marks)

f) Atomic spectrometry is applicable to most gas phase elements over a wide range of concentrations and involves detecting, measuring and analyzing radiation that is either absorbed or emitted from from the atoms or ions of the element. Briefly comment on the principle of atomic absorption spectrometry and atomic fluorescence spectrometry as techniques of atomic spectrometry. (6 marks)

QUESTION TWO (20 MARKS)

a) State the function of a mass analyzer and name two types which are used in mass spectroscopy. (4 marks)

b) Chromatography is one of the main techniques used in separation of a mixture into individual components. Factors other than boiling points of compounds will affect separation. Name and explain four factors that affect separation of compounds while using gas chromatography mass spectroscopy as a technique. (8 marks)

c) Atomic absorption spectroscopy is a technique used to determine the level of contaminants in food and environmental samples. For reliable results, calibration is critical. Define calibration and briefly explain single-level calibration and multilevel calibration.

(8 marks)

QUESTION THREE (20 MARKS)

a) State the Beer-Lambert law. (2 marks)

b) Various atoms in a molecule may be regarded as balls of different masses and the covalent bonds between them as weightless tiny springs holding such balls together. Atoms in a molecule are not still but they vibrate. The two types (modes) of fundamental molecular vibrations known are: (a) stretching and (b) bending vibrations (deformations). Explain the working of two types of each mentioned modes of vibration. (4 marks)

c) Explain the following terms as used in nuclear magnetic resonance spectroscopy. (4 marks)

i. Chemical shift

ii. TMS

iii. Downfield shift

iv. Spin-spin coupling

d) Infrared spectroscopy deals with the recording of the absorption of radiations in the infrared region of the electromagnetic spectrum. Draw a schematic diagram of a double-

beam infrared spectrometer and explain the functions of the radiation source, sample (reference cells) and photometer. (6 marks)

e) Spectrometric instruments and separation instruments are widely applied in laboratory analytical instrumentation both in developing and developed countries. Give four applications of Nuclear Magnetic Resonance as one of the equipment widely used in the developed countries. (4 marks)

QUESTION FOUR (20 MARKS)

a) Using an example in each case, briefly explain how the concept of fluorescence, phosphorescence and chemiluminescence works under photophysical processes. (6 marks)

b) The wavelength of a violet light is 400 nm. Calculate its frequency and wave number. (4 marks)

c) Since atoms cannot vibrate or rotate as a molecule does, when energy is absorbed, electronic transitions take place and are seen as sharp lines as opposed to broad bands or peaks in the molecular spectra. These lines are specific for the element being studied and contain information on its atomic structure. If energy is provided to an atom, there are three possible transitions. State and explain the three possible transitions occurring in atoms. (6 marks)

d) Differentiate between split and splitless techniques of sample injection in a Gas liquid chromatography technique. (4 marks)

QUESTION FIVE (20 MARKS)

a) Name any three detectors used for the measurement of intensity of transmitted light. (3 marks)

b) The reactions which are caused by heat and in absence of light are called thermal reactions. Excited molecules due to absorption of light produce photochemical reactions. Give five differences between photochemical reactions and thermochemical reactions. (5 marks)

c) Calculate the energy associated with an infrared having wavelength 286 mm. Give the answer in kcal/mol and also in kJ/mol. (6 marks)

d) Briefly explain the working of a flame atomic absorption spectrometry and Graphite furnace atomic absorption spectrometry. (6 marks)