BOWEN UNIVERSITY IWO, OSUN STATE
COLLEGE OF AGRICULTURE, ENGINEERING AND SCIENCE
INDUSTRIAL CHEMISTRY PROGRAMME
2022/2023 SESSION B.SC DEGREE FIRST SEMESTER EXAMINATION

Course Code: CHM 315 **Course Title:** Separation Methods and Analysis
Date: Monday, 13/02/2023 **Credit Unit:** 3 **Time Allowed:** 2½ Hours

Instruction: Answer any TWO (2) questions from each section.

USEFUL CONSTANTS
***h* = 6.62 x 10^{-34} J sec**
***c* = 3.00 x 10^{8} m/sec**
R = 1.097 × 10^{7} m^{-1} or 1.097x10^{5} cm^{-1}

SECTION A

QUESTION ONE

(a) What is chromatography? (2 marks)
(b) Describe the different types of chromatography based on:
  i. mobile and stationary phases (2 marks)
  ii. contact between mobile and stationary phases (2 marks)
  iii. interaction between solute and stationary phases (2 marks)
(c) A chromatographic separation of four compounds gave the following results,

Analyte	1	2	3	4
Signal	0.072	0.046	0.061	0.01
Retention Time	120 sec	255 sec	310 sec	608 sec
Peak Width at base	28 sec	70 sec	55 sec	98 Ec

Given that the dead time (t_m) 16.7 sec,

i. what are the retention factors for analyte 1 and 4? (3 marks)
ii. what are the number of plates for the column based on analyte 4? (2 marks)
iii. what is the resolution factor for analytes 3 and 4? (3 marks)
iv. what is the resolution factor for analytes 1 and 2? (3 marks)
v. What can you say about the separation between analytes 1&2 and 3&4? (1 mark)

QUESTION TWO

(a) What is the significance of selectivity factor in chromatography? (2 marks)

(b) In a chromatographic analysis of lemon oil, a peak for limonene has a retention time of 8.36 min with a baseline width of 0.96 min. γ-Terpinene elutes at 9.54 min with a baseline width of 0.64 min. What is the resolution between the two peaks? (4 marks)

(c) In a chromatographic analysis of low molecular weight acids, butyric and isobutyric acid elute with a retention time of 7.63 and 5.98 min respectively. Given that the column's void time is 0.31min calculate the selectivity factor for butyric acid and isobutyric acids. (6 marks)

(d) In spectroscopy, what is the difference between mechanical slit width and spectral width? (4 marks)

(e) Explain why single beam optics are good for emission spectroscopy while double beam optics are more suitable for absorption spectroscopy. (4 marks)

QUESTION THREE

(a) Differentiate between absorption and emission spectroscopy. Give some examples of each type of spectroscopy (4 marks)

(b) Describe the different motions of atoms or molecules that exist in absorption or emission spectroscopy (3 marks)

(c) State the mathematical expressions for the Lambert law, Beer's law and Beer-lambert law and define all the term in the expression (4 marks)

(d) Palladium reacts with Thio-Michler's ketones forming a coloured 1:4 complex. A 0.20ppm Pd sample gave an absorbance of 0.390 at 520nm using a 1.00cm cell. Calculate the molar absorptivity (ϵ) for the palladium Thio-Michler ketone complex. Molecular weight of Pd is 106.4 (4 marks)

(e) Briefly describe the basic components of spectroscopic equipment used in studying either absorption or emission spectroscopy (5 marks)

SECTION B

QUESTION ONE

(a) What is spectroscopy? (2 marks)

(b) Highlight the important applications of spectroscopy. (2 marks)

(c) Give the fundamental formula of spectroscopy. Define the terms. (3 marks)

(d) State two key difference between atomic spectroscopy and molecular spectroscopy. (2 marks)

(e) Briefly describe the spectrum of atomic hydrogen. Using an illustrative diagram, explain Bohr's model of the hydrogen atom showing some of the jumps that give rise to absorption (jumps up) and emission (jumps down). (6 marks)

(f) Calculate the wavelength of the visible line in the hydrogen spectrum that has the longest wavelength. What is the energy difference between the two levels of the atom involved in the transition giving rise to this line? (5 marks)

QUESTION TWO

(a) Explain in general terms the mechanism in a prism and grating that leads to the attainment of monochromatic radiation. What are the advantages of a grating over a prism? (6 marks)

(b) Explain the significance of the slit width of a monochromator. What is the advantage(s) of making the slit width smaller? What is the disadvantage(s) of making the slit width smaller?
(4 marks)

(c) Define electromagnetic radiation. What is the relationship between the energy (E) and frequency (υ) of electromagnetic radiation? What is the relationship between the energy and wavelength (λ) of electromagnetic radiation? (3 marks)

(d) Write the types of radiation observed in the electromagnetic spectrum going from high to low energy. Also include what types of processes occur in atoms or molecules for each type of radiation. (3 marks)

(e) In analytical chemistry, spectroscopy is used in two primary ways. List and explain. (4 marks)

QUESTION THREE

(a) Use a schematic diagram of a simple atomic absorption spectroscopy (AAS) to explain the principle of AAS. Explain the dependent factor responsible for AAS measurement.
(6 marks)

(b) List the type of dissolution that are necessary in preparation of a solid sample for an AAS analysis. (4 marks)

(c) With examples, explain the feature which makes microwave dissolution suitable for sample preparation. (2 marks)

(d) Outline the procedures involved in carrying out determination of the concentration of elements by AAS. (6 marks)

(e) Distinguish between wet ashing and dry ashing. (2 marks)