



NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY

FACULTY OF APPLIED SCIENCES

DEPARTMENT OF APPLIED CHEMISTRY

CHROMATOGRAPHIC SEPARATIONS

SCH 4292

Second Semester Examination Paper

May 2016

This examination paper consists of 4 pages

Time Allowed: 3 hours

Total Marks: 100

Examiner's Name: Dr. A. Maringa

INSTRUCTIONS

- a) Answer ALL questions in section A and any three (3) questions in section B
- b) Each question in section A carries 10 marks and each question in section B carries 20 marks

MARK ALLOCATION

QUESTION	MARKS
SECTION A: 1.	10
2.	10
3.	10
4.	10
SECTION B: 5	20
6	20
7	20
8	20
TOTAL POSSIBLE MARKS	100

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SECTION A

1. a) Name the five types of liquid chromatography and give the details of the separation mechanism of each. [10 marks]

2. a) What important property of supercritical fluids is related to their densities? [2 marks]
b) The retention time for a mixture component A is 5.4 min, and the retention time for a mixture component B is 3.3 min. The retention time for the solvent is 1.1 min. What is the selectivity for component A relative to B? [3 marks]
c) What advantages does a thermal conductivity detector have over a flame ionization detector? [5 marks]

3. Define the following terms:
 - i. Isocratic elution.
 - ii. Gel filtration.
 - iii. Elution.
 - iv. Chromatogram.
 - v. Stationary phase. [10 marks]

4. a) State the advantages of TLC over HPLC. [3 marks]
b) Describe the steps involved in paper chromatography. [7 marks]

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SECTION B

5. a) List the variables that lead to band broadening in gas chromatography. [3 marks]
- b) Explain how the temperature programming feature of most modern GCs can be useful in separating complex mixtures. [5 marks]
- c) Contrast the packed column and the open-tubular capillary column (of GC) in terms of design, diameter, length, how the stationary phase is held in place, ability to resolve complex mixtures, and amount of sample injected. [12 marks]
6. Discuss the advantages and disadvantages of the following HPLC detectors.
- i. UV absorption detector.
 - ii. Refractive index.
 - iii. Fluorescence.
 - iv. Conductivity.
 - v. LC-IR. [20 marks]
7. Substances A and B have retention times of 16.40 and 17.63 min, respectively, on a 30.0-cm column. An unretained species passes through the column in 1.30 min. The peak widths (at base) for A and B are 1.11 and 1.21 min, respectively. Calculate (a) the column resolution, (b) the average number of plates in the column, (c) the plate height, (d) the length of column required to achieve a resolution of 1.5, and (e) the time required to elute substance B on the column that gives an R_s value of 1.5. [20 marks]

8. a) Define internal standard. [2 marks]
- b) Compare and contrast the advantages and disadvantages of the UV absorbance detector vs. the refractive index detector. [5 marks]
- c) Explain why an internal standard is important in a quantitative analysis by GC. Also state what is plotted on the x- and y-axes when plotting the standard curve in internal standard procedures. [5 marks]
- d) A standard solution containing 6.3×10^{-8} M iodoacetone and 2.0×10^{-7} M p-dichlorobenzene (an internal standard) gave peak areas of 395 and 787, respectively, in a gas chromatogram. A 3.00 mL unknown solution of iodoacetone was treated with 0.100 mL of 1.6×10^{-5} M p-dichlorobenzene and the mixture was diluted to 10.00 mL. Gas chromatography gave peak areas of 633 and 520 for iodoacetone and p-dichlorobenzene, respectively. Find the concentration of iodoacetone in the 3.00 mL of original unknown. [8 marks]

End of question paper!!!!

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