



NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY

FACULTY OF APPLIED SCIENCES

DEPARTMENT OF APPLIED CHEMISTRY

CHROMATOGRAPHIC SEPARATIONS

SCH 4292

Supplementary Examination Paper

July 2016

This examination paper consists of 4 pages

Time Allowed: 3 hours

Total Marks: 100

Examiner's Name: Dr. A. Maringa

INSTRUCTIONS

1. Answer ALL questions in section A and any three (3) questions in section B
2. 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) One type of chromatography separates small molecules from large ones. Name this type and tell how such a separation occurs. [2 marks]
- (b) How does partition chromatography differ from adsorption chromatography? [4 marks]
- (c) Differentiate between the use of a cation exchange resin and an anion exchange resin in terms of whether the charged sites are positive or negative and whether cations or anions are exchanged. [4 marks]

2. (a) What is meant by temperature programming in GC? [2 marks]
- (b) Two chromatography peaks show retention times of 1.3 and 2.5 min. The width at the base of the first peak is 0.25 min, and the width at the base of the second peak is 0.29 min. What is the resolution? [3 marks]
- (c) The retention time of a certain component on a particular 2.0 m gas chromatography column is 3.1 min. The width at the base for the peak is 0.39 min. How many theoretical plates are in this column and what is the height equivalent to a theoretical plate? [5 marks]

3. Define the following terms:
 - i. Selectivity factor
 - ii. Dead time or void time
 - iii. Normal phase packing
 - iv. Ion pair chromatography
 - v. Guard column [10 marks]

4. (a) Why must HPLC mobile phases and samples be filtered prior to use in the HPLC system? [2 marks]
- (b) Distinguish between isocratic elution and gradient elution. [2 marks]
- (c) For supercritical carbon dioxide, predict the effect that the following changes will have on the elution time in an SFC experiment.
- Increase the flow rate (at constant temperature and pressure).
 - Increase the pressure (at constant temperature and flow rate).
 - Increase the temperature (at constant pressure and flow rate). [6 marks]

SECTION B

5. (a) What is the purpose of derivatization in chromatography? [2 marks]
- (b) List the variables that lead to band separation in gas-liquid chromatography. [3 marks]
- (c) Explain the use of a guard column and in-line filters. [5 marks]
- (d) What do the letters GC-MS stand for? Briefly describe how the GC-MS detector works and why it is such a useful detector. [10 marks]
6. (a) Nonpolar aromatic compounds were separated by HPLC on an octadecyl (C₁₈) bonded phase. The eluent was 65 vol% methanol in water.
- How would the retention times be affected if 90% methanol were used instead?
 - Octanoic acid and 1-aminooctane were passed through the same column described in part (a), using an eluent of 20% methanol/80% buffer (pH 3.0). State which compound is expected to be eluted first and why.
Octanoic acid (CH₃CH₂CH₂CH₂CH₂CH₂CH₂CO₂H)
1-Aminooctane (CH₃CH₂CH₂CH₂CH₂CH₂CH₂CH₂NH₂) [5 marks]

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(b) What are the general steps in developing an isocratic separation for reversed-phase chromatography with one organic solvent? [7 marks]

(c) Discuss the advantages of open tubular columns over packed columns in gas chromatography. [8 marks]

7. Discuss the advantages and disadvantages of gas chromatography. [20 marks]

8. The following data are for a liquid chromatographic column:

Length of Packing	24.7 cm
Flow rate	0.313 mL/min
V_M	1.37 mL
V_S	0.164 mL

A chromatogram of a mixture of species A, B, C, and D provided the following data:

	Retention time (min)	Width of Peak Base, W (min)
Nonretained	3.1	-
A	5.4	0.41
B	13.3	1.07
C	14.1	1.16
D	21.6	1.72

Calculate for A, B, C, and D the retention factor and the distribution constant. [20 marks]

End of question paper!!!!

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