



**NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY**

**FACULTY OF APPLIED SCIENCES**

**DEPARTMENT OF APPLIED CHEMISTRY**

**APPLICATIONS OF ANALYTICAL CHEMISTRY – SCH 4106**

**Special Paper**

**2025**

This Examination Paper consists of 4 pages

**Time Allowed:** 3 hours

**Total Marks:** 100

**Special Requirement:** Calculator

**Internal Examiner:** Dr. A. Maringa

**External Examiner:** Prof. G. Mehlana

**INSTRUCTIONS**

1. Answer ALL questions in Section A and any three (3) questions in Section B
2. Questions in Section A carries 40 marks and each question in Section B carries 20 marks

**MARK ALLOCATION**

<b>QUESTION</b>	<b>MARKS</b>
1.	40
2.	20
3.	20
4.	20
5.	20
<b>TOTAL POSSIBLE MARKS</b>	<b>100</b>

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**SCH 4106**

## SECTION A

1. (a). Explain briefly:
- (i) why  $\text{HNO}_3$ , rather than other acids is most commonly used for acid digestion? [3 marks]
  - (ii) Why HF digestion is used for samples containing silicates? [2 marks]
- (b). Dry ashing is a relatively simple method of removing the organic matter that can be used for relatively large samples and requires little of the analyst's time. In the open vessel method, the sample is placed in a suitable crucible and is ignited in a muffle furnace.
- (i) Mention any 3 materials that used to make the crucibles. [3 marks]
  - (ii) Beside volatilization, what other drawbacks are encountered in dry ashing. [2 marks]
- (c). The selectivity and efficiency of the LLE process is mainly dependent on the choice of the two immiscible solvents. The extraction efficiency depends on a number of factors. Mention any 5 factors that affect the extraction efficiency. [5 marks]
- (d). A combination of nitric acid and sulphuric acid is a good dehydrating and oxidizing mixture. One advantage of the nitric acid-sulphuric acid combination is that it can used at high temperatures and this will lead to rapid oxidation. Give the disadvantages of the mixture. [5 marks]
- (e). In preparing the chromatography paper, a baseline is drawn in pencil for the positioning the spots. Why is ink never used for drawing the baseline? [4 marks]

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- (f). A chromatographic analysis for the chlorinated pesticide Dieldrin gives a peak with a retention time of 8.68 min and a baseline width of 0.29 min. What is the number of theoretical plates? Given that the column is 2.0 m long, what is the height of a theoretical plate in mm? [6 marks]
- (g). Methanol and ethanol are separated in a capillary GC column with retention times of 370 and 385 s, respectively, and half widths ( $w_{1/2}$ ) of 9.42 and 10.0 s. An unretained butane peak occurs at 10.0 s. Calculate the separation factor and the resolution. [10 marks]

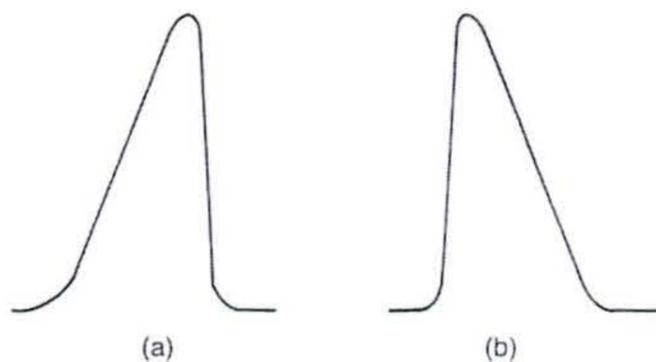
## SECTION B

2. (a). What problem may occur when a sample is stored? [3 marks]
- (b). Describe the factors to consider when preparing a sample for analysis. [5 marks]
- (c). Microwave assisted extraction (MAE) method was used to extract semi volatile organic compounds. Describe the procedure involved and highlight the advantages and disadvantages of MAE. [12 marks]
3. (a). A solute has a  $K_D$  between water and chloroform of 5.00. Suppose we extract a 50.00 mL sample of a 0.050 M aqueous solution of the solute with 15.00 mL of chloroform.
- (i). What is the extraction efficiency? [5 marks]
- (ii). What volume of chloroform must we use to extract 99.9% of the solute? [5 marks]
- (iii). Determine the extraction efficiency for two extractions. [5 marks]
- (iv). Determine the number of extractions required to ensure that we extract 99.9% of the solute. [5 marks]

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4. (a). One of the most common solvent used in HPLC is acetonitrile. Suggest the positive features of acetonitrile that makes it desirable. [10 marks]

(b). Peak fronting or peak tailing as shown in the Fig. below are typical examples of unsymmetrical peaks.



Peaks exhibiting (a) fronting and (b) tailing.

Suggest the probable cause and cure of peak fronting and peak tailing. [10 marks]

5. Although the mechanism governing separation in HPLC and TLC are identical, differences in practical aspects lead to us to compare and contrast the performance of HPLC and TLC. Compare and contrast the performance of HPLC and TLC. [20 marks]

**END OF QUESTION PAPER**