



TECHNICAL UNIVERSITY OF MOMBASA

FACULTY OF APPLIED AND HEALTH SCIENCES

DEPARTMENT OF PURE & APPLIED SCIENCES

UNIVERSITY EXAMINATION FOR:

MASTER OF SCIENCE IN CHEMISTRY

ACH 5112: MODERN TRENDS IN ANALYTICAL METHODS

END OF SEMESTER EXAMINATION

SERIES: DECEMBER 2016

TIME: 3 HOURS

DATE: Pick Date Jan 2017

Instructions to Candidates

You should have the following for this examination

-Answer Booklet, examination pass and student ID

This paper consists of **SIX** Question(s). Answer any **FOUR** questions.

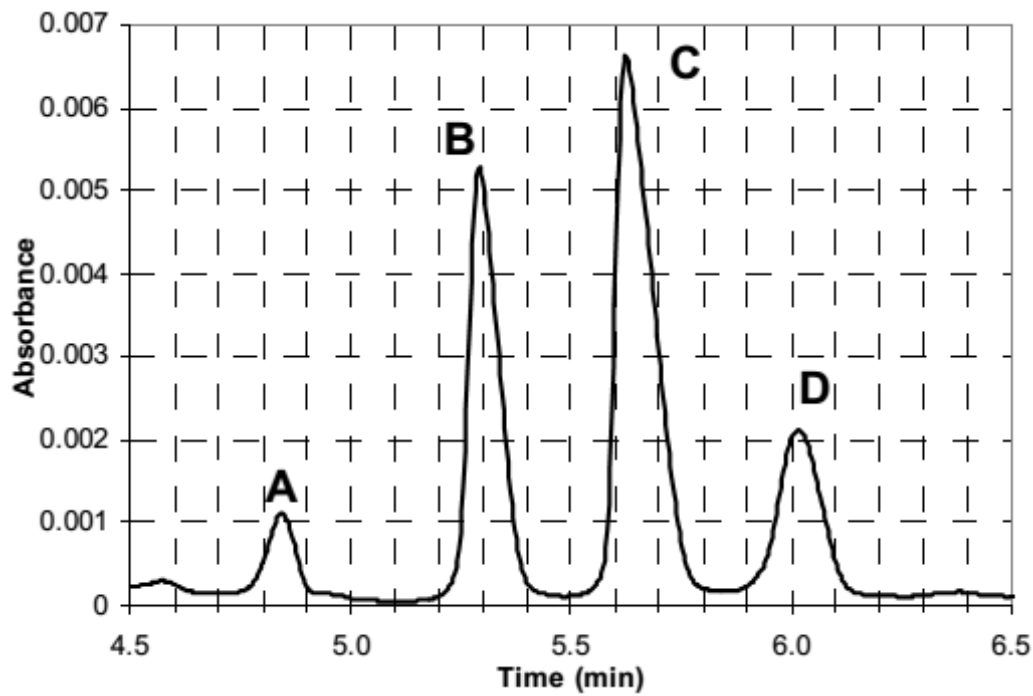
Do not write on the question paper.

Question ONE

- (a) Explain the three terms in the van Deemter equation $HETP = A + B/u + Cu$, and how they contribute to band broadening in a chromatographic separation. Indicate how the effects of the main factors can be minimized. (18 marks)
- (b) Account for the terms that contribute to band-broadening in CE, HPLC and GC separations. (7 marks)

Question TWO

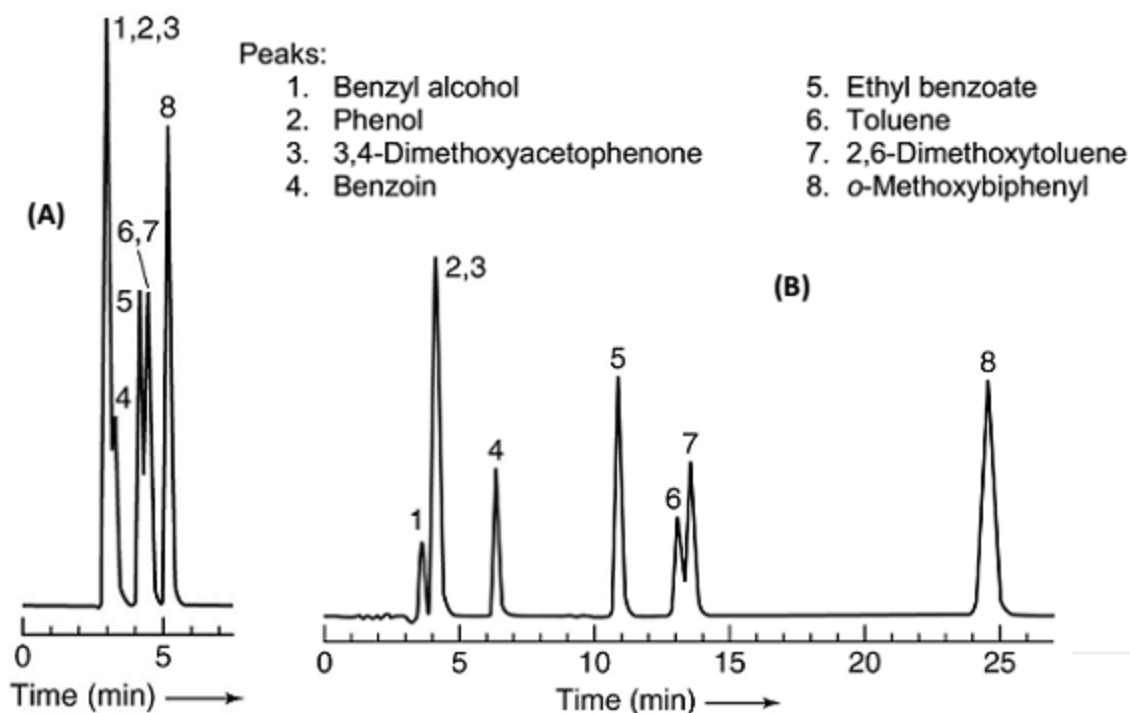
- (a) Describe the principles of operation of Solid-phase extraction (SPE) and solid phase microextraction (SPME). Indicate advantages and disadvantages for each. (18 marks)
- (b) The chromatogram below was obtained for a reverse-phase HPLC separation on a 25 cm column, using UV absorbance detection. Un-retained compounds elute in 0.15 minutes.



- (i) Calculate the number of theoretical plates for component B. (2 marks)
- (ii) Calculate the resolution of compounds C and B. (2 marks)
- (iii) Which compound is the most polar? Justify your choice. (3 marks)

Question THREE

- (a) Describe the mechanism of separation in capillary zone electrophoresis (CZE). Indicate the parameters that can be changed to optimize separation conditions in CZE? (12 marks)
- (b) The two chromatograms A and B, shown below were recorded using reverse phase HPLC, using the same column, with a water/methanol mobile phase.



- (i) Explain how the separation could be changed from chromatogram A to B. (4 marks)
- (ii) Give an example of a detector that might be used for these compounds. (1 mark)
- (iii) Estimate the theoretical plate height, H , and the number of theoretical plates, N , for peak 8 of chromatogram B. The length of the column was 15.0 cm. (Estimate the values of the variables required for the calculation from the axes of the chromatogram). (4 marks)
- (iv) Estimate and comment on the resolution for peaks 6 and 7 given $t_{R6} = 13.2$ min, $t_{R7} = 13.7$ min, $\omega_6 = 0.8$ min and $\omega_7 = 1.0$ min. (4 marks)

Question FOUR

- (a) Describe the following ionization techniques as applied in Mass Spectrometry, indicating them as either hard or soft.
 - (i) Electron impact (EI) (5 marks)
 - (ii) Electrospray ionization (ESI) (7 marks)
 - (iii) Chemical ionization (CI). (6 marks)
- (b)
 - (i) Outline the basic structure of a Linear Quadrupole mass analyzer (4 marks)
 - (ii) Describe how the Linear Quadrupole mass analyzer functions. (3 marks)

Question FIVE

- (a) Outline the structure, principles of operation and application for the following detectors, and further classify each one of them as a concentration or mass detector.
- (i) Flame ionization detector (FID) (8 marks)
 - (ii) Electron capture detector (ECD) (8 marks)
- (b) Carbon dioxide is the most popular mobile phase used in supercritical fluid chromatography. Give any FOUR reasons why it is superior to other mobile phases. Describe how its solvating power and polarity can be modified? (6 marks)
- (c) Provide any TWO advantages and ONE disadvantage of use of supercritical CO₂ in extraction. (3 marks)

Question SIX

- (a) (i) State why Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) is the preferred method of determining rare earth elements, such as Cerium, rather than the use of optical detection,? (6 marks)
- (ii) Describe any TWO advantages and TWO types of interferences associated with ICP-MS. (8 marks)
- (b) Describe briefly the following techniques in capillary electrophoresis separation and application.
- (i) Micellar Electrokinetic Chromatography (MEKC) (6 marks)
 - (ii) Capillary Electrochromatography (CEC) (5 marks)