



MACHAKOS UNIVERSITY

University Examinations 2020/2021 Academic Year
SCHOOL OF PURE AND APPLIED SCIENCES
DEPARTMENT OF PHYSICAL SCIENCES
FIRST YEAR SECOND SESSION EXAMINATION FOR
MASTER OF SCIENCE (CHEMISTRY)
SCH 800: ADVANCED SEPARATION TECHNIQUES

DATE: 27/8/2021

TIME: 8.30-10.30 AM

INSTRUCTIONS:

- The paper consists of **two** sections.
- Section **A** is **compulsory**.
- Answer any **two** questions from section **B**.

SECTION A – COMPULSORY (30 MARKS)

QUESTION ONE (30 MARKS)

- a) Define each of the following terms as used in analytical separation techniques;
- i) Gradient elution. (1 mark)
 - ii) Isocratic elution. (1 mark)
- b) Differentiate between the following terms
- i) Reversed Phase Solid phase extraction (SPE) and Normal Phase SPE (2 marks)
 - ii) Laminar flow and turbulent flow. (2 marks)
 - iii) Gas solid chromatography and Gas liquid Chromatography. (2 marks)
- c) Suggest a type of compound that would be suitable for the separation on the following phase types of solid phase extraction. (4 marks)
- i) Octadecyl bonded, end-capped silica
 - ii) Quaternary amine bonded silica with Cl^- counterion
 - iii) Silica gel with no bonded phase
 - iv) Resin-based packing

- d) Substances A and B were found to have retention times of 16.40 and 17.63, respectively, on 30.0 cm column. An unretained species passed through the column in 1.30 min. The peak widths (at base) for A and B were 1.11 min and 1.21 min, respectively. Calculate:
- i) The column resolution. (1.5 marks)
 - ii) The average number of plates in the column. (2 marks)
 - iii) The plate height. (2 marks)
 - iv) The length of the column required to achieve a resolution of 1.5. (3 marks)
 - v) The time required to elute substance B on the longer column. (2 marks)
- e) Solid phase extraction (SPE) is an increasingly useful sample preparation technique. With SPE, many of the problems associated with liquid/liquid extraction can be prevented, such as incomplete phase separations, less-than-quantitative recoveries, use of expensive, breakable specialty glassware, and disposal of large quantities of organic solvents. Briefly explain the five-step method development process as applied in SPE. (7.5 marks)

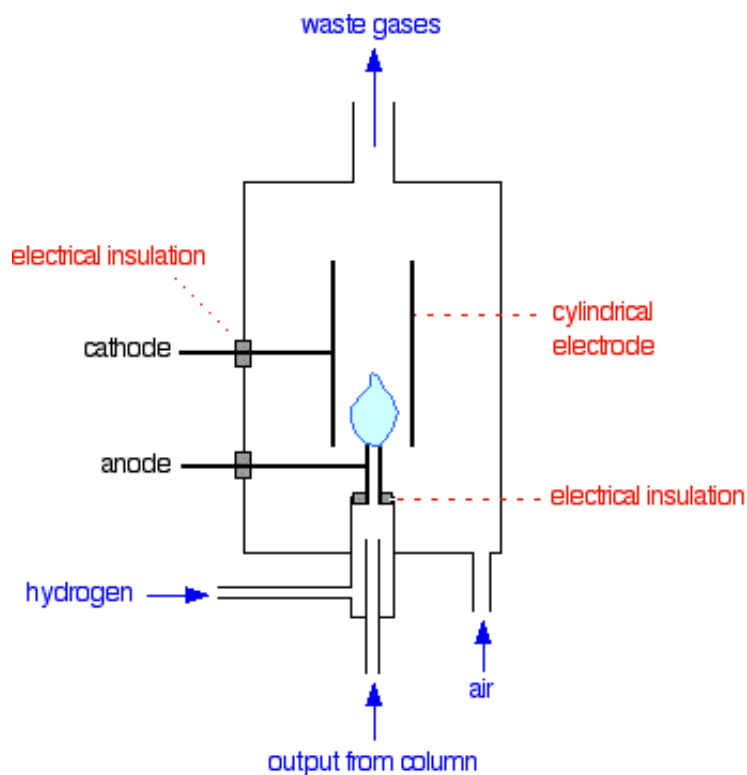
SECTION B: ANSWER ANY TWO QUESTIONS (15 MARKS EACH)

QUESTION TWO (15 MARKS)

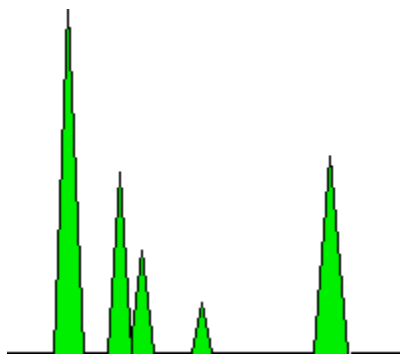
- a) Briefly explain the application of the Snyder solvent triangle application method as used in liquid chromatography. (2 marks)
- b) Dozens of detectors have been investigated and used during the development of gas chromatography (GC). Briefly explain four (4) characteristics of an ideal detector for sample detection using GC. (4 marks)
- c) An analytical chemistry student working as an intern in a chemical manufacturing company was given the following environmental samples: Sulphur containing compounds and halogen containing compounds. Using a gas chromatographic technique and with a relevant detector, describe the methodology that you will use to determine the concentration of halogen in the sample. (5 marks)
- d) Briefly explain:
- i. The difference between a concentration-sensitive detector and mass flow- sensitive detector. (2 marks)
 - ii. The effect of increasing the weight of a stationary phase relative to the packing weight on the plate height of a GC column. (2 marks)

QUESTION TWO (15 MARKS)

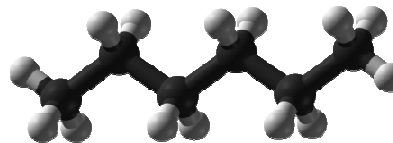
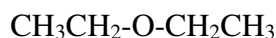
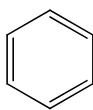
- a) One form of detector that is used in chromatography is a flame ionization detector (FID). This diagram below shows a flame ionization detector. Explain:



- How the FID works. (5 marks)
 - Why the detector is kept at a temperature hotter than the temperature of the column. (1 mark)
 - Why it is often useful to divert some of the output from a chromatography column to a mass spectrometer to get a fragmentation pattern, which can help you to identify what is coming out at that time. However, this can't be done if one uses flame ionization detection. (2 marks)
- b) The output from the detector is recorded as a series of peaks as shown in this simplified diagram. What information can you get from the areas under the peaks? (2 marks)



- c) What is the order in which the following compounds would be eluted from an HPLC column containing a reversed-phase packing? Explain your answer. (3 marks)
- Benzene, diethyl ether, n-hexane.



- d) List two (2) advantages of gradient runs over isocratic runs as used in HPLC. (2 marks)

QUESTION FOUR (15 MARKS)

- a) Define the following terms as used in Flow Injection Analysis:
- i) Dispersion coefficient. (1 mark)
 - ii) Critical temperature of a gas. (1 mark)
- b) Describe three (3) major differences between non-segmented continuous flow analysis and flow injection analysis. (3 marks)
- c) Discuss briefly the five (5) instrumentation factors that must be considered when constructing a flow injection system. (5 marks)
- d) Grignard reaction can be used to form triphenylmethanol. Using relevant equations where necessary, explain how the desired product can be separated from the mixture of by-products and residual starting material. (5 marks)

QUESTION FIVE (15 MARKS)

- a) Explain the difference between split mode and splitless mode as used in gas chromatography. (2 marks)
- b) An insect pheromone (a chemical) is being extracted from beetles by shaking a flask containing 1.00 g of coarsely ground beetles with 10 mL of ethyl acetate. This solvent is filtered and saved, and then a second 10 mL of ethyl acetate is added to the filtered beetle solids for a second extract.
- i. Explain the purpose of the second batch of ethyl acetate. (2 marks)
 - ii. List three methods that can be used to improve the percent of pheromone extracted. (3 marks)

- c) Different compounds have different retention times. For a particular compound separated using Gas liquid Chromatography, briefly explain three factors that will influence the retention time of an analyte. (3 marks)
- d) Suppose that the partition coefficient for an amine, B, is $K = 3$ and the acid dissociation constant of BH^+ is $K_a = 1 \times 10^{-9}$. If 50 mL of 0.01 M aqueous amine is extracted with 100 mL of solvent, what will be the formal concentration remaining in the aqueous phase at:
- pH 10.02 (3 marks)
 - pH 8.01 (2 marks)