



MACHAKOS UNIVERSITY

University Examinations 2018/2019

SCHOOL OF PURE AND APPLIED SCIENCES

DEPARTMENT OF PHYSICAL SCIENCES

FIRST YEAR FIRST SEMESTER EXAMINATION FOR

MASTER SCIENCE (CHEMISTRY)

SCH 800: ADVANCED SEPARATION TECHNIQUES

DATE: 12/4/2019

TIME: 10.00-1.00 PM

INSTRUCTIONS:

- The paper consists of **two** sections.
- Section **A** is **compulsory** (30 marks).
- Answer any **two** questions from section **B** (each 20 marks).

QUESTION ONE (COMPULSORY) (30 MARKS)

- a) Non chromatographic separation technique which is actually solvent-solvent extraction (partition) is one of the necessary steps in the analysis of an analyte in analytical techniques
- State three purposes which solvent extraction serves. (3 marks)
 - State clearly the principle underlying the solvent-solvent extraction (2 marks)
 - State Nernst distribution law in the solvent-solvent extraction defining each term in the equation. (2 marks)
 - What is the difference between partition (distribution) coefficient and distribution ratio? (2 marks)
 - Define the separation factor for two solutes undergoing solvent-solvent extraction. (2 marks)
- b) Briefly explain the principles behind each of these methods of solvent extraction listed below
- Chelate extraction (4 marks)
 - Extraction by solvation (4 marks)

- iii. Extraction by pair ion formation (4 marks)
- c) Explain briefly the following steps in the solvent-solvent extraction procedure
 - i. Stripping (3 marks)
 - ii. Salting-out agents (2 marks)
 - iii. Use of masking agents (2 marks)

QUESTION TWO (20 MARKS)

- a) In chromatographic separations, there are normally two phases, stationary and mobile. Unlike the solvent-solvent separation techniques, chromatography can also be used as an analytical technique for identification and quantitative.
 - i. Differentiate between adsorption chromatography and partition chromatography with the help of chemical equations. (2 marks)
 - ii. Define retention time t_R as used in chromatography (2 marks)
 - iii. Define retention volume V_R (2 marks)
 - iv. Describe what height equivalent to a theoretical plate (HETP) means in the efficiency of separation of components in a mixture by a chromatographic column. (2 marks)
- b) In gas chromatography the stationary phase is either a solid or a liquid adsorbed on a solid base.
 - i. Explain why a solid phase stationary has limited use in gas chromatography (2 marks)
 - ii. The solid support in the gas liquid chromatography which supports the stationary liquid phase also causes tailing of peaks. Explain why tailing occurs. (2 marks)
 - iii. Explain how silanization helps in the prevention of the tailing of peaks mentioned in b(ii). (3 marks)
 - iv. Differentiate between splitter flow and non-splitter flow in relation to the injection port as applied to gas chromatography. (3 marks)
 - v. Explain the properties of organic compounds which can be analyzed by gas Chromatography. (2 marks)

QUESTION THREE (20 MARKS)

- a) There are two types of gas chromatography detectors based on (i) responses proportional to the concentration of the sample and another type based on the rate at which the sample is delivered to the sensing element.

- i. Describe briefly how the thermal conductivity detector (TCD) functions mentioning the suitable carrier gases used. (6 marks)
- ii. Describe how the electron capture detector functions outlining only salient features mentioning the suitable carrier gases. (6 marks)
- b) i The flame ionization detector functions on a different principle from those a(i) and(ii) above. Describe how it functions. (5 marks)
- ii Describe the role of temperature programming in the GC analysis of a mixture of compounds. (3 marks)

QUESTION FOUR (20 MARKS)

- a) High performance liquid chromatography (HPLC) normally operates at a lower temperature than GC (between freezing point and boiling point) of the solvent. Two types of columns are normally incorporated in the HPLC known respectively as normal phase and reverse phase.
 - i. What kind of compounds can be separated by liquid chromatography? (3 marks)
 - ii. Describe briefly and differentiate between normal phase and reverse phase columns in HPLC. (4 marks)
 - iii. Differentiate between isocratic elution and gradient elution. (3 marks)
 - iv. Explain why gradient elution is ideal in the separation of complex mixtures of compounds with a wide range of polarities. (2 marks)
- b) Describe briefly the ion-exchange chromatography. (4 marks)
- c) Describe briefly the Gel Permeation (Filtration) chromatography. (4 marks)

QUESTION FIVE (20 MARKS)

- a) Supercritical fluid chromatography (SFC) is considered to be an intermediate chromatography whose properties are said to lie between gas and liquid chromatography. Define the following terms as used in SFC.
 - i. Supercritical fluid. (2 marks)
 - ii. Critical pressure (P_c). (2 marks)
 - iii. Critical temperature (T_c). (2 marks)
- b) Show the phase diagram of pressure-temperature of a pure substance under critical conditions by plotting pressure versus temperature. (3 marks)
- c) Of all the mobile phases that have been used in SFC, CO_2 is the most successful and widely used.

- i. State four disadvantages of SFC due to mainly mobile phase and equipment related. (2 marks)
 - ii. State what is sub-critical fluid which is sometimes used in SFC. (2 marks)
 - iii. State clearly two advantages of SFC over both GC and LLC. (2 marks)
- d)
 - i Describe briefly the principles in capillary electrophoresis. (3 marks)
 - ii Briefly discuss where capillary electrophoresis finds application. (2 marks)