



KENYATTA UNIVERSITY

UNIVERSITY EXAMINATIONS 2010/2011

INSTITUTIONAL BASED PROGRAMME (IBP) – AUGUST SESSION

EXAMINATION FOR THE DEGREE OF MASTER OF SCIENCE

SCH 500: ADVANCED SEPARATION TECHNIQUE AND DATA ANALYSIS

DATE: Friday, 30th December, 2011

TIME: 9.00 a.m. – 12.00 p.m.

INSTRUCTIONS:

Answer **ALL** questions.

Statistical tables are attached.

For rejection test use only **Q**

Question One

- a) Analytical chemistry is a branch of chemistry that is multi disciplinary.
 - i) Use an example to explain this statement. (1 mark)
 - ii) Hence or otherwise briefly describe the step of “sampling” in chemical analysis. (2 marks)
- b) “Serial standards are prepared by diluting the stock solution”
 - i) Describe the preparation of the standard stock solution (1 mark)
 - ii) Explain ‘calibration’ using the serial standards (2 marks)
 - iii) State an importance of the calibration curve (1 mark)
 - iv) Address the problem of ‘linearity’ of the calibration curve (2 marks)
 - v) Express the best line of fit given a centroid of **(60., 13.1)** and a slope of 2.09 (1 mark)
- c) Differentiate briefly between the following:-
 - i) Elution isothermally and by temperature programming

- ii) Detection limit and sensitivity of an instrument
- iii) At-distribution and z-distribution
- iv) Blocked and un-blocked experimental designs
- v) GSC and GLC

(5 marks)

Question Two

- a) Analytical separations are important for both qualitative and quantitative determinations
 - i) Give an example to support this statement (2 marks)
 - ii) Explain how separation is achieved by partition in solvent extraction and in chromatography (4 marks)
 - iii) Describe masking in solvent extraction (1 mark)
 - b) List two for each of the following:-
 - i) characteristics of columns
 - ii) Modes of injection ports
 - iii) Properties of a supercritical fluid
 - iv) Precautions during spotting
 - v) Ways of improving sensitivity of detection in electrophoresis
- (5 marks)
- c) Describe the chromatogram in terms of:-
 - i) Resolution
 - ii) Quantification
 - iii) Affinity for the stationary phase
 - iv) HETP
- (5 marks)
- d) Using block diagrams, differentiate between:-
 - i) Components of a GLC and HPLC equipment
 - ii) Laminar flow and pressure driven flows
 - iii) A distribution curve for random errors and for systematic errors.

(6 marks)

Question Three

- a) Write brief notes on the working of the following detectors
- i) UV-detector
 - ii) Thermal conductivity detector
- (4 marks)
- b) State the Nerst law, and describe how a weak acid (HBz) deviates from the law
- (3 marks)
- c) What is the importance of the following:
- i) Paired-test
 - ii) Blank analysis
 - iii) Capillary electrophoresis
 - iv) Flow injection analysis
 - v) Confidence limit of the mean
 - vi) Retardation factor
- (6 marks)
- d) Show the advantage of batch extraction of a metal ion (M^+) distributing between 50ml of water and two successive extractions of 25ml chloroform ($K_d = 5.0$)
- (3 marks)

Question Four

- a) Write a brief explanation as a quality control manager to advice the production section to 'stop' the process of manufacturing a foam product whose density should be 250 kg/L.
- (6 marks)
- b) Replicate chromatograms were run for a sample containing 2 chlorobenzene derivatives X and Y, by SFC and GLC. The data for X is given below:
- | | | | | | | | |
|------------|-------------|---|-----|-----|-----|-----|-----|
| SFC column | Rt | : | 4.5 | 4.7 | 4.6 | 4.4 | |
| | Peak height | : | 150 | 152 | 149 | 150 | |
| GLC column | Rt | : | 6.1 | 6.3 | 6.0 | 6.5 | 8.2 |
| | Peak height | : | 182 | 179 | 180 | 184 | 183 |
- i) Did SFC significantly reduce the MEAN retention times? (8 marks)

- ii) Check consistency of replicate values of peak height between the two methods. (3 marks)
- c) Draw an arrow for EOF and label the ions in the capillary tube to indicate separation by Charge, size and electrophoretic mobility.

