UNIVERSITY OF SWAZILAND

FACULTY OF HEALTH SCIENCES

Department of Environmental Health sciences

Supplementary examination 2015

Title : Instrumental methods for environmental analysis

Code : EHM 212

Time : 2 hours

Marks : 100

Instructions:

1. Answer any 4 questions,
2. Each question weighs 25 marks,
3. Start each question on a fresh page,
4. Non-programmable scientific calculators may be used,

Additional material;

- Graph paper,

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QUESTION 1

a) Define the following terms as applied in analytical chemistry, giving mathematical expressions where applicable;

i) Capillary action
ii) Retention volume
iii) Internal standard
iv) Separation factor
v) Phase ratio [5]

b) What assumptions are made in the application of the following techniques;

i) Standard additions, [2]
ii) Linear least squares method, [3]

c) The phosphorus content in a urine sample was analysed by employing a spectrophotometric method. The data for the standards and sample are given below:

Standard	**1**	**2**	**3**	**4**	**Urine sample**
P (mg/L)	1.00	2.00	3.00	4.00	X
Absorbance	0.205	0.410	0.615	0.820	0.625

Employ the least squares regression method to:

i) **Calculate** the slope, intercept and concentration of phosphorus in the urine sample. [10]
ii) Plot the best straight, i.e. the best least square line. [5]

QUESTION 2

a) Define an external standard calibration and the ideal property that the standard is expected to have. [2]

b) A solution containing 3.47 mM of an analyte and 1.72 mM of a standard gave peak areas of 3.473 and 10.222, respectively, in a chromatographic analysis. Then 1.00 mL of 8.47 mM of the standard was added to 5.00 mL of unknown analyte, and the mixture was diluted to 10.0 mL. The solution gave peak areas of 5.428 and 4.431 for the unknown analyte and the standard, respectively;

i) Calculate the response factor for the analyte. [3]
ii) Find the concentration of the standard (mM) in the 10.0 mL of mixed solution. [4]
iii) Find the concentration of analyte (mM) in the 10.0 mL of mixed solution. [3]
iv) Find the concentration of the analyte in the original unknown. [3]

c) Solute X is 12 times more soluble in trichloromethane than in water. What mass of solute X will be extracted from 1.00 L aqueous solution containing 25.0 g by shaking with 100 mL of dichloromethane? [5]

d) The partition coefficient of butanedioic acid between ethyoxyethane and water is 5. What mass of butanedioic acid will be extracted when a solution containing 5.00g butanedioic acid in 100 mL of water is shaken with 10 mL of ethyoxyethane? [5]

QUESTION 3

a) Give the main advantages of Gas Chromatography (GC) over other separation techniques. [5]

b) Give the ideal properties of a good GC detector [5]

c) Draw a fully labelled schematic diagram of a GC. [5]

d) What are the important considerations for liquid stationary phase materials used in GLC? [5]

e) The retention time, t_r, of a solute is 25.0 s on a column with $N = 5.4 \times 10^3$. Calculate;

   i) $W_{1/2}$ (width at half the peak height) [3]

   ii) W, the expected base width of the peak. [2]

QUESTION 4

With reference to Thin Layer Chromatography (TLC);

a) Give four (4) things that TLC can achieve. [4]

b) Give an example or name of a process for each of the following as used in TLC:

   i) A stationary phase,

   ii) Solid support on which the stationary phase in mounted,

   iii) Mechanism of separation of solute between the mobile and stationary phases. [3]

c) Briefly describe the procedure (steps) for the development of a chromatogram and the detection of analyte spots. [7]

During the analysis of samples using a GC, the following are obtained;

d) Two chromatograms C_1 and C_2 (for each compound). Explain the significance of N (number of theoretical plates), during the separation if N_1 is greater than N_2. [3]

e) A chromatogram with two peaks (correspondingto C_1 and C_2). Explain the significance of t_R (retention time), during the separation if t_{R1} is less than t_{R2}. [3]

f) Two chromatograms of the same mixture from two different GC instruments. How would you determine which instrument gives better separation? [5]

QUESTION 5

a) For the wall coated tubular column used in GC, diagrammatically illustrate its main structural features. [5]

b) Give three types of detectors used in a GC. For each type discuss:
   i) Its function,
   ii) Its operation,
   iii) Its desirable properties. [15]

c) Define column efficiency, as used in GC. Explain how column efficiency is affected by the plate height and the number of theoretical plates. [5]