



UNIVERSITY OF SWAZILAND

FACULTY OF HEALTH SCIENCES

Department of Environmental Health sciences

Supplementary examination 2013/14

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:
- i) Solvent extraction
 - ii) Capillary action
 - iii) Retention volume
 - iv) Resolution
 - v) Separation factor
 - vi) Phase ratio [6]
- b) What assumptions are made in the application of the following techniques
- i) Standard additions [2]
 - ii) Linear least squares method [2]
- c) Describe the procedure for the solvent extraction of a solute from a 50 mL aqueous sample, using 100 mL carbon tetrachloride as the organic phase. [5]
- d) The partition coefficient of Arsenite chloride (AsCl_3) between ether and 6 M HCl acid is 0.54. How many times must one extract 50 cm³ of 6 M HCl acid containing As with 20 cm³ portions of ether in order to remove 98 % of the As? [5]
- e) Give the main advantages of Gas Chromatography (GC) over other separation techniques. [5]

QUESTION 2

- a) Define an external standard calibration and the ideal property that the standard is expected to have. [2]
- b) 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]
- c) To determine the concentration of Cr in soil, six standard solutions of Cr^{6+} were prepared and the necessary colouring agents added. A UV-vis spectrophotometer was used to measure the absorbance for each solution at a particular wavelength. The results are in the table below. The unknown was measured to have an absorbance of 0.418 and the blank, 0.003 absorbance units.

Solution Number	Concentration (mg/L)	Absorbance (a.u.)
1	0	0.003
2	1	0.078
3	2	0.163
4	4	0.297
5	6	0.464
6	8	0.600

- i) Define a reagent blank? [1]
- ii) The absorbance reading for the reagent blank above is not characteristic of a blank. Explain why the blank reading is not zero and how this effect is corrected for the experiment. [3]
- iii) Determine the concentration of the unknown using the graphical method. [4]

QUESTION 3

With respect to Gas Chromatography (GC),

- a) Draw a fully labelled schematic diagram of a GC. [6]
- b) Give the ideal properties of a good detector [5]
- c) What are the important considerations for liquid stationary phase materials used in GLC? [5]
- d) 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]
- e) A solute was eluted completely from a chromatographic column over 2 mins, 40 sec. Calculate its retention volume if its flow rate is 20 mL/min. [4]

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 is 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]
- d) For the phrase: ' R_f value',

- i) Give its meaning. [1]
- ii) What is the mean of a low R_f value versus a high R_f value during separation of polar compounds from a mixture? [3]
- iii) Use a diagrammatical illustration to show how it can be determined. [5]
- iv) Give two (2) factors that influence the R_f value of a compound. [2]

QUESTION 5

- a) For the wall coated tubular column used in GC, diagrammatically illustrate its main structural features. [5]
- b) For the electron capture detector (ECD) used as a GC detector, discuss,
 - i) Its function,
 - ii) The factors determining its choice,
 - iii) Its desirable properties. [5]
- 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]
- d) With respect to the solvent extraction of metals,
 - i) Define a chelating agent. [1]
 - ii) Write the equation for the formation of a metal chelate (complex), and identify the reactants and products. [3]
 - iii) Give two (2) properties of a metal chelate formed from the solvent extraction of a metal as a metal chelate. [2]
- e) Define the following figures of merit (FOM) as applied in data assurance protocols;
 - i) Sensitivity,
 - ii) Detection limits (DL),
 - iii) Linear dynamic range,
 - iv) Limits of quantitation (LOQ) [4]