UNIVERSITY OF SWAZILAND

FACULTY OF HEALTH SCIENCES

Department of Environmental Health sciences

Main examination 2015

Title : Instrumental methods for environmental analysis

Code : EHM 212

Time : 2 hours

Total Marks : 100

Instructions:

1. This paper contains 5 printed pages (including the cover page) with a total of 5 questions,
2. Question 1 is compulsory,
3. Answer any other 3 questions,
4. Each question weighs 25 marks,
5. Start each question on a fresh page,
6. Drawings, schematics and graphs must be large and well labelled,
7. Non-programmable scientific calculators may be used,
8. No table of scientific constants is provided.

Additional material:

- Graph paper (1)

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QUESTION 1
COMPULSORY

A mining company is suspected of disposing effluent waste that contains the pollutant metal Chromium (VI), into a river. You are employed as an environmental chemist for the SEA and tasked with determining if the hypothesized concentrations are within the legal framework. The concentration of the metal in the waste stream was then determined using voltammetry where the peak height of the current signal is proportional to concentration. A standard addition method was performed by adding specific volumes of 10 mg/LCr(VI) solution to the sample as shown in the table below. All solutions were made up to a final volume of 20 mL. The peak currents obtained from the analyses are also tabulated below.

Volume of sample (mL)	Volume of std(mL)	Peak current (μA)	Blank sample
10	**0**	**8**	**0.09**
10	**2**	**16**	**0.07**
10	**5**	**25**	**0.08**
10	**10**	**41**	**0.09**

a) Using the method of linear squares, plot the 'best straight line' using the data above as far as possible. [12]

b) Hence, determine the concentration of Cr(VI) contained in the **ORIGINAL** sample. [3]

c) Determine the strength of the correlation between the variables plotted above.[3]

d) Calculate the standard deviation of the slope and intercept, given that, $Sy_{/x}$ = 0.4329. [4]

e) Calculate the detection limits for the analysis. [3]

QUESTION 2

a) Define the following terms:
   i) Solvent extraction
   ii) Limits of quantification
   iii) Dynamic range
   iv) Limits of linear responses
   v) Matrix matching [5]

b) Give two (2) scenarios when the standard additions method is preferred over normal calibration methods. [2]

c) Iron (Fe) was analysed in a zinc electrolyte. The signal obtained from an Atomic absorption spectrometer (AAS) was 0.381 absorbance units. 5 mL of a 0.2 M Fe standard was added to 95 mL of the sample. The signal obtained was 0.805. Calculate the concentration of Fe in the original sample. [5]

d) The partition coefficient of Arsenite chloride ($AsCl_3$) between ether and 6 M HCl acid is 0.44. How many times must one extract 60 cm^3 of 6 M HCl acid containing As with 20 cm^3 portions of ether in order to remove 98 % of the As? [5]

e) Using an appropriate expression, show how the solvent extraction of a metal as a chelate is affected by solution pH. [3]

f) Briefly explain the procedure for the solvent extraction of a solute from a 50 mL aqueous sample using 100 mL carbon tetrachloride. [5]

QUESTION 3

The following functional groups are arranged in order of increasing polarity;

$-CH=CH_2$, -X, -OR, -CHO, $-CO_2R$, $-NR_2$, $-NH_2$, -OH, $-CONR_2$, $-CO_2H$.

a) A mixture of organic compounds containing the above functional groups is to be separated using Thin Layer Chromatography (TLC). Briefly describe, with illustrations where possible, the procedure for the separation of an ink mixture that contains $-CH=CH_2$, –CHO and $–CO_2H$ using. In your discussion show or explain the following points;

   i) Suggest a possible stationary phase that can be used to achieve the separation. [1]
   ii) Briefly outline the steps to be undertaken to achieve the separation. [7]

b) With reference to TLC;

   i) Give four (4) things that TLC can achieve. [4]
   ii) What is the meaning of 'R_f value'? [1]

iii) What is the meaning of a low R_f value versus a high R_f value during separation of polar compounds from a mixture? [3]

iv) Use a schematic diagram to illustrate how this value can be experimentally determined. [4]

v) Give two (2) factors that influence the R_f value of a compound. [2]

vi) Define resolution and give two (2) ways in which it can be improved. [3]

QUESTION 4

a) Define the following terms or acronyms as applied in Gas Chromatography (GC);
   i) Selectivity factor
   ii) Column resolution
   iii) Distribution constant
   iv) HETP
   v) Retention volume [5]

b) In GC, analyte separation occurs in the columns.
   i) Explain why temperature programming is important [1]
   ii) Explain the structural differences between open tubular and packed columns, with labeled illustrations where possible. [6]
   iii) What are the advantages of open tubular columns to packed columns? [4]
   iv) Hence, explain why open tubular columns provide better sensitivity than packed columns. [4]

c) Briefly outline the properties of a good mobile phase in GC, giving an example. [5]

QUESTION 5

In chromatographic analyses, the actual separation occurs in the column.

a) Give two of the most common mechanisms responsible for the separation. [2]

b) Calculate the minimum distribution coefficient that allows 99.9% of a solute extraction from 50 mL of water with:
   i) Two 25.0 mL extracted with n-hexane,
   ii) Five 10 mL extracted with n-hexane [6]

c) During the chromatographic analysis of a sample on a 30 cm long column, 2 adjacent peaks, A and B, appear with the following properties;

Component	t_R (min)	W (min)
A	8.36	0.96
B	9.54	0.64

i) Calculate the resolution between A and B. [4]

ii) If the retention time for an unretained solute is 2.20 min, calculate the selectivity factor for A and B. [4]

iii) Calculate the capacity factors for both A and B. [4]

iv) Calculate the average number of plates in the column. [3]

v) Hence, determine the plate height. [2]