UNIVERSITY OF SWAZILAND

FACULTY OF HEALTH SCIENCES

Department of Environmental Health sciences

Main 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. Drawings, schematics and graphs must be large and well labelled,
5. Non-programmable scientific calculators may be used,

Additional material:

- Graph paper,

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QUESTION 1

a) Define the following terms;
   i) Liquid-liquid extraction
   ii) Reagent blank
   iii) Internal standard
   iv) Chromatography
   v) Matrix effect [5]

b) Give 5 physical properties that a good extraction solvent should possess. [5]

c) Solute A has a partition coefficient of 3 between benzene and water. Suppose that 100 mL of a 0.01 M aqueous solution of A is extracted with benzene. What percent fraction of A remains in solution if;
   i) One 500 mL extraction is performed, [3]
   ii) Five (5) extractions using 100 mL are performed? [3]
   iii) Determine the amount (moles) of solute remaining in solution after extraction with the 500 mL benzene. [6]

d) Describe **briefly** how a mixture of sugar in vegetable oil can be separated. [3]

QUESTION 2

A 3.12 g stick of chewing gum is ashed at high temperatures, dissolved in 5mL of concentrated nitric acid and then diluted to the mark in a 100 mL volumetric flask. To 25 mL volumetric flasks are added a 5.00 mL aliquot of the dissolved chewing gum sample and an addition of 300.0 ppm aluminium. After dilution to volume, the solution is analysed for aluminium by graphite furnace atomic absorption at 309 nm. The data are shown in the following table.

Vol. added standard, mL	Signal
0	0.214
0.1	0.386
0.2	0.554
0.3	0.728

a) i) Use the method of least squares **to obtain the best straight line** for the calibration curve. [10]
   ii) **Graphically** determine the concentration of aluminium in the original gum in μg/g. [4]
   iii) Determine the strength of the correlation between the signal and the amount of standard added. [4]

b) 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]

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

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) 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 Thin Layer Chromatography (TLC). In your discussion show or explain the following points;

i) The solvent front, [1]
ii) The origin, [1]
iii) Common types of solid support, [3]
iv) The stationary phase, [1]
v) The orientation of the spots after separation, [3]
vi) Common methods for identifying the separated compounds. [2]

b) Explain the important mechanisms responsible for the isolation/separation of compounds in chromatography. [6]

c) With reference to TLC;

i) What is the meaning of 'R_f value'? [1]
ii) Use a schematic diagram to illustrate how this value can be experimentally determined. [4]
iii) 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) SCOT
iii) GSC
iv) HETP
v) Retention volume
vi) Elution [6]

b) In GC, analyte separation occurs in the columns.

i) Explain why the column is housed in a temperature programmable oven. [2]

ii) Explain the differences between open tubular and packed columns, with labeled illustrations where possible. [6]

iii) Give an example of an adsorbent used as a packing in GC columns. [1]

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

d) An unretained solute passes through a chromatography column in 3.7 min and the analyte requires 8 min. Calculate the adjusted retention time and the capacity factor for the analyte. [5]

QUESTION 5

a) With respect to validation of an analytical method, define the following terms;

i) Limits of detection,

ii) Linearity range,

iii) Limits of quantification,

iv) Percentage recovery [4]

b) The distribution ratio for palladium (II) chloride between 3M HCl and tri-n-butyl phosphate (TBP) is 2.3. How many times must 15 mL of a 5.0×10^{-3} M solution of $PdCl_2$ be extracted with fresh 5.00 mL portions of TBP in order to remove 99.5 % of the metal? [5]

c) In GC, substances A and B have retention times of 16.40 and 17.63 min, respectively, on a 30 cm column. An unretained species passes through the column in 1.30 min. the peak widths at base for A and B are 1.11 and 1.21 min, respectively. Calculate;

i) The column resolution, [2]

ii) The average number of plates, [4]

iii) The plate height, [2]

iv) The length of column required to achieve a resolution of 1.5, [4]

v) The time required to elute substance B on the column that gives an R_s value of 1.5.[4]