

**UNIVERSITY OF SWAZILAND**  
**Faculty of Health Sciences**  
**DEGREE IN ENVIRONMENTAL HEALTH**  
**MAIN EXAMINATION PAPER 2013**

**TITLE OF PAPER** : Instrumental Methods for Environmental Health

**COURSE CODE** : EHM 212

**DURATION** : 2 HOURS

**MARKS** : 100 MARK

**INSTRUCTIONS** :

- : READ THE QUESTIONS & INSTRUCTIONS CAREFULLY
- : ANSWER ANY FOUR QUESTIONS
- : EACH QUESTION CARRIES 25 MARKS
- : WRITE NEATLY & CLEARLY
- : NO PAPER SHOULD BE BROUGHT INTO NOR OUT OF THE EXAMINATION ROOM
- : BEGIN EACH QUESTION ON A SEPARATE SHEET OF PAPER

**Addition Material:**

1. Graph paper
2. Periodic Table

**DO NOT OPEN THIS QUESTION PAPER UNTIL PERMISSION IS GRANTED BY THE INVIGILATOR**

### QUESTION 1

- a) Define the following terms;
- i) Solvent extraction
  - ii) Organic phase
  - iii) Internal standard
  - iv) Retention time,  $t_R$ ,
  - v) Matrix matching [5]
- b) Give 5 physical properties that a good extraction solvent should possess. [5]
- c) The pain reliever phenacetin is soluble in cold water to the extent of 0.90g in 1310 mL and soluble in diethyl ether to an extent of 1.0g in 90 mL.
- i) Determine the approximate distribution coefficient for phenacetin in those 2 solvents. [3]
  - ii) If 140 g of phenacetin were dissolved in 100 mL of distilled water, how much ether would be required to extract 95 % phenacetin in a single extraction? [5]
  - iii) What percentage of the phenacetin would be extracted from the aqueous solution in part (ii) above by two 50 mL portions of ether? [3]
- d) Draw and label the schematic of a Gas Chromatogram (GC) [4]

### QUESTION 2

- a) 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]
- b) During the chromatographic analysis of a sample, 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]
- c) With reference to Thin Layer Chromatography (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]
- d) In solvent extraction, the solubility of different compounds in a mixture can be altered into their water soluble forms. Briefly describe how you would change the solubility of the following groups of compounds:

- i) Carboxylic acids, [2]
- ii) Phenols. [2]

### QUESTION 3

- a) Differentiate between an internal and an external standard. [2]
- b) A spectrophotometric technique was used to determine protein content in goat's milk. Prepared working standards were determined together with the unknown sample and the results are shown below.

Test Tube	Concentration (mg/L)	Signal output
1	0.8	0.9
2	0.6	0.45
3	0.4	0.22
4	0.2	0.11
5	0	0
6	x	0.34

- i) Determine the concentration (mg/L) of the unknown sample using the graphical method, hence determine the protein content. [8]
  - ii) Briefly explain what you would perform the analysis if there was evidence of matrix effects which interfered with the output signal. [5]
  - iii) What is the appropriate name given to sample 5, and how is it different from the other samples? [3]
- c) Briefly describe the procedure for the extraction of a solute dissolved in 50 mL of an aqueous phase with 100 mL of carbon tetrachloride. [7]

### QUESTION 4

- a) Aliquots of a standard solution of an element X were mixed with an unknown sample containing X for AAS analysis. The standard solution contained 1.00mg of X per liter. The following absorbance readings were obtained.

Vol. of unknown (mL)	Vol. of standard (mL)	Total volume (mL)	Absorbance
10	0	100.00	0.163
10	1.00	100.00	0.240
10	2.00	100.00	0.319

10	3.00	100.00	0.402
10	4.00	100.00	0.478

- i) Determine the concentration of each standard in mg/L. [5]
- ii) Determine the concentration of X in the unknown sample. [10]
- b) With reference to Thin Layer Chromatography (TLC);
- i) Briefly describe the procedure for the development of a chromatogram and the detection of analyte spots. [7]
- ii) Give 3 advantages of TLC over paper chromatography. [3]

### QUESTION 5

- a) With reference to Gas Chromatography (GC), briefly discuss;
- i) The main features of open and tubular columns, [6]
- ii) The main advantages of open tubular columns over packed columns, [4]
- iii) The functions and ideal properties of the solid support and stationary phase, [5]
- iv) The important property and example of a mobile phase. [2]
- b) For the ECD GC detector discuss,
- i) Its function,
- ii) The factors determining its choice,
- iii) Its desirable properties. [4]
- c) i) What is a chelating agent? [1]
- ii) Write an equation for the formation of a metal chelate (complex) and identify the reactant and product. [3]

# PERIODIC TABLE OF ELEMENTS

PERIODS	GROUPS																		
	I	IIA	III	IV	V	VI	VII	VIII	VIII	VIII	IX	X	XI	XII	XIII	IVA	VA	VIA	VIIA
1	IA 1 H	IIA 2 He	TRANSITION ELEMENTS																VIIIA 18 Ar
2	Li 3	Be 4	TRANSITION ELEMENTS																Ne 10
3	Na 11	Mg 12	TRANSITION ELEMENTS																Ar 18
4	K 19	Ca 20	TRANSITION ELEMENTS																Kr 36
5	Rb 37	Sr 38	TRANSITION ELEMENTS																Xe 54
6	Cs 55	Ba 56	TRANSITION ELEMENTS																Rn 86
7	Fr 87	Ra 88	TRANSITION ELEMENTS																

*Lanthanide Series		*Actinide Series	
La 57	Ce 58	Th 90	Pa 91
Pr 59	Pm 61	U 92	Uu 93
Nd 60	Sm 62	Np 94	Am 95
Pm 61	Eu 63	Pu 96	Cm 97
Sm 62	Gd 64	Bk 97	Cf 98
Eu 63	Tb 65	Ce 98	Es 99
Gd 64	Dy 66	Am 99	Fm 100
Tb 65	Ho 67	Eu 100	Md 101
Dy 66	Er 68	Am 101	No 102
Ho 67	Tm 69	Am 102	Lr 103
Er 68	Yb 70		
Tm 69	Lu 71		

( ) indicates the mass number of the isotope with the longest half-life.

Atomic mass -  
Symbol -  
Atomic No.