UNIVERSITY OF SWAZILAND
FIRST SEMESTER EXAMINATION 2010/2011

TITLE OF PAPER	**:**	**Instrumental Methods For Environmental Analysis - II**
COURSE NUMBER	**:**	**EHS 574**
TIME ALLOWED	**:**	**Two(2) Hours**
INSTRUCTIONS	**:**	**Answer any four (4) questions. Each Question carries 25 marks.**

A periodic table and other useful data have been provided with this paper.

You are not supposed to open this paper until permission to do so has been granted by the Chief Invigilator.

Question 1(25 marks)

(a) Explain the following terms as briefly as possible:
  (i) Elution
  (ii) Volume flow rate, F
  (iii) Retention time, t_R
  (iv) Adjusted retention time, t^1_R
  (v) Relative retention, α [5]

(b) Explain the term 'chromatogram' Using an illustrative diagram show how it it is employed for both the identification and the quantitation of a solute during a GC analysis. [6]

(c) For each of the following set of terms, give an expression that relates them.
  (i) Retention Volume, V_R, retention time, t_R and Volume flow rate of the mobile phase, F.
  (ii) Capacity factor, k, retention time t_R and dead time, t_M [2]

(d) What is 'Temperature programming' of a chromatographic column? [2]

(e) Discuss the effects of the following on the efficiency of a chromatographic column.
  (i) Increasing the column temperature.
  (ii) Using the temperature programming method. [7]
  (iii) Use illustrative diagrams to demonstrate the effects in (b) [3]

Question 2(25 marks)

(a) State the Nernst's distribution law. Give the mathematical expression for it and define all the parameters involved in it. [3]

(b) Enumerate the differences between distribution coefficient, K_D and distribution ratio, D, used during solvent extraction analysis. Illustrate this difference with an example. [4]

(c) For the extraction of a weak acid, HB, whose anion (B^-), does not penetrate the organic phase and is monomeric in both phases:
  (i) **State** the expression for its distribution ratio, D, and define all the parameters in it [3]
  (ii) Give two of the factors that influence the value of D. [2]

(d) The distribution ratio, D, of a solute being extracted from water with carbon tetrachloride was 85.0

(i) Calculate the percentage of the solute extracted from the aqueous phase when 50.0mLof 1.0×10^{-3}M aqueous solution of the solute is extracted with 50.0mL of carbon tetrachloride? [8]

(ii) Would you have preferred employing two successive extractions, each with 25.0mL carbon tetrachioride? Justify your answer. [5]

Question 3(25 marks)

(a) For the 'Gas Liquid Chromatograph (GC):

(i) Draw and label its schematic diagram. [4]

(ii) What is the basic requirement for a mobile phase material? Give four commonly employed examples. [3]

(iii) State the functions and four ideal properties each of the stationary phase (the liquid phase) and the solid support. [8]

(iv) Give four commonly employed detectors, stating whether they are universal or selective respectively. [4]

(b) Mention the various sectors of the society where chromatographic methods have been employed as analytical tools. Give four industries or research laboratories in Swaziland where these methods are being routinely used. [6]

Question 4(25 marks)

(a) What is 'column efficiency' in gas chromatography? How is its value influenced by 'loading' of the column, N (the number of theoretical plates), and H (the plate height)? What other factors influence it? [8]

(b) (i) What are the advantages/disadvantages of open tubular columns over packed columns used for GC analysis. Briefly account for the difference.

(ii) Give two structural differences between them. [7]

(c) During the chromatographic analysis of a mixture of chlorinated pesticides, in which a 2.0 m long column was used, a peak with a retention time, t_r, of 8.68 min. and a baseline width of 0.36 min.. was identified to be dieldrin.

(i) Estimate N and H for this column. [6]

(ii) Calculate the capacity factor, k, for dieldrin if the dead time, t_m, for the column is 0.30 min. [2]

(iii) An adjacent peak to that of dieldrin has a retention time, tr, of 9.76 min and a baseline width of 0.62 min. Calculate the resolution between the two peaks. [2]

Question 5(25 marks)

(a) State three advantages of thin layer chromatography (TLC) over paper chromatography. [3]

(b) With respect to TLC.

(i) Identify two examples each of the stationary phases and mobile phases commonly employed for analysis. [4]

(ii) What stationary phases would you employ for the analysis of:

• a polar compound

• a weakly polar compound [2]

(c) Describe the procedure for chromatogram development and detection of analyte spots during TLC. [7]

(d) (i) Define 'R_f-Value' for a solute during TLC analysis. [1]

(ii) Using a diagrammatic illustration only demonstrate how it can be measured for a given solute. [4]

(e) Identify four factors that influence the R_f Value of a compound. [4]

THE PERIODIC TABLE OF ELEMENTS

NON-METALS

METALLOIDS

METALS

Group	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
	IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIIIB			IB	IIB	IIIA	IVA	VA	VIA	VIIA	VIIIA
Period 1	1 **H** 1.008																	2 **He** 4.003
2	3 **Li** 6.94	4 **Be** 9.01											5 **B** 10.81	6 **C** 12.01	7 **N** 14.01	8 **O** 16.00	9 **F** 19.00	10 **Ne** 20.18
3	11 **Na** 22.99	12 **Mg** 24.31											13 **Al** 26.9	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.06	17 **Cl** 35.45	18 **Ar** 39.9
4	19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.90	23 **V** 50.94	24 **Cr** 52.01	25 **Mn** 54.9	26 **Fe** 55.85	27 **Co** 58.71	28 **Ni** 58.71	29 **Cu** 63.54	30 **Zn** 65.37	31 **Ga** 69.7	32 **Ge** 72.59	33 **As** 74.92	34 **Se** 78.96	35 **Br** 79.91	36 **Kr** 83.80
5	37 **Rb** 85.47	38 **Sr** 87.62	39 **Y** 88.91	40 **Zr** 91.22	41 **Nb** 91.22	42 **Mo** 95.94	43 **Tc** 98.9	44 **Ru** 101.1	45 **Rh** 102.9	46 **Pd** 106.4	47 **Ag** 107.9	48 **Cd** 112.4	49 **In** 114.8	50 **Sn** 118.7	51 **Sb** 121.8	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
6	55 **Cs** 132.9	56 **Ba** 137.3	71 **Lu** 174.9	72 **Hf** 178.5	73 **Ta** 180.9	74 **W** 183.8	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.1	79 **Au** 196.9	80 **Hg** 200.6	81 **Tl** 204.4	82 **Pb** 207.2	83 **Bi** 208.9	84 **Po** 210	85 **At** 210	86 **Rn** 222
7	87 **Fr** 223	88 **Ra** 226.0	103 **Lr** 257	104 **Unq**	105 **Unp**	106 **Unh**	107 **Uns**	108 **Uno**	109 **Une**									

Lanthanides	57 **La** 138.9	58 **Ce** 140.1	59 **Pr** 140.9	60 **Nd** 144.2	61 **Pm** 146.9	62 **Sm** 150.9	63 **Eu** 151.3	64 **Gd** 157.3	65 **Tb** 158.9	66 **Dy** 162.5	67 **Ho** 164.9	68 **Er** 167.3	69 **Tm** 168.9	70 **Yb** 173.0
Actinides	89 **Ac** 227.0	90 **Th** 232.0	91 **Pa** 231.0	92 **U** 238.0	93 **Np** 237.1	94 **Pu** 239.1	95 **Am** 241.1	96 **Cm** 247.1	97 **Bk** 249.1	98 **Cf** 251.1	99 **Es** 254.1	100 **Fm** 257.1	101 **Md** 258.1	102 **No** 255

*Numbers **below** the symbol indicates the **atomic masses**; and the numbers above the symbol indicates the atomic numbers.*

General data and fundamental constants

Quantity	Symbol	Value
Speed of light†	c	$2.997\,924\,58 \times 10^{8}$ m s^{-1}
Elementary charge	e	$1.602\,177 \times 10^{-19}$ C
Faraday constant	$F = eN_A$	9.6485×10^{4} C mol^{-1}
Boltzmann constant	k	$1.380\,66 \times 10^{-23}$ J K^{-1}
Gas constant	$R = kN_A$	8.314 51 J K^{-1} mol^{-1}
		$8.205\,78 \times 10^{-2}$ dm^{3} atm K^{-1} mol^{-1}
		62.364 L Torr K^{-1} mol^{-1}
Planck constant	h	$6.626\,08 \times 10^{-34}$ J s
	$\hbar = h/2\pi$	$1.054\,57 \times 10^{-34}$ J s
Avogadro constant	N_A	$6.022\,14 \times 10^{23}$ mol^{-1}
Atomic mass unit	u	$1.660\,54 \times 10^{-27}$ kg
Mass of		
electron	m_e	$9.109\,39 \times 10^{-31}$ kg
proton	m_p	$1.672\,62 \times 10^{-27}$ kg
neutron	m_n	$1.674\,93 \times 10^{-27}$ kg
Vacuum permeability†	μ_0	$4\pi \times 10^{-7}$ J s^{2} C^{-2} m^{-1}
		$4\pi \times 10^{-7}$ T^{2} J^{-1} m^{3}
Vacuum permittivity	$\varepsilon_0 = 1/c^2\mu_0$	$8.854\,19 \times 10^{-12}$ J^{-1} C^{2} m^{-1}
	$4\pi\varepsilon_0$	$1.112\,65 \times 10^{-10}$ J^{-1} C^{2} m^{-1}
Bohr magneton	$\mu_B = e\hbar/2m_e$	$9.274\,02 \times 10^{-24}$ J T^{-1}
Nuclear magneton	$\mu_N = e\hbar/2m_p$	$5.050\,79 \times 10^{-27}$ J T^{-1}
Electron g value	g_e	2.002 32
Bohr radius	$a_0 = 4\pi\varepsilon_0\hbar^2/m_e e^2$	$5.291\,77 \times 10^{-11}$ m
Rydberg constant	$R_\infty = m_e e^4/8h^3 c\varepsilon_0^2$	$1.097\,37 \times 10^{5}$ cm^{-1}
Fine structure constant	$\alpha = \mu_0 e^2 c/2h$	$7.297\,35 \times 10^{-3}$
Gravitational constant	G	$6.672\,59 \times 10^{-11}$ N m^{2} kg^{-2}
Standard acceleration of free fall†	g	9.806 65 m s^{-2}

† Exact (defined) values

Prefixes

f	p	n	μ	m	c	d	k	M	G
femto	pico	nano	micro	milli	centi	deci	kilo	mega	giga
10^{-15}	10^{-12}	10^{-9}	10^{-6}	10^{-3}	10^{-2}	10^{-1}	10^{3}	10^{6}	10^{9}