UNIVERSITY OF SWAZILAND

Faculty of Health Science

Department of Environmental Health Sciences

Main Examination 2011

Title of paper: INSTRUMENTAL METHODS FOR ENVIRONMENTAL ANALYSIS

Course code: EHS 574

Time allowed: 2 HOURS

Marks allocation: 100 Marks

Instructions:

1) Answer **Four** questions
2) Each question is weighted 25 marks
3) Write neatly and clearly
4) Begin each question in a separate sheet of paper

This paper is not to be opened until the invigilator has granted permission

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

Question 1(25 marks)

(a) Briefly explain the following terms
- (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) What is a chromatogram? With an illustrative diagram show how the chromatogram is employed for both qualitative and quantitative analysis of a sample. [6]

(c) Give an expression that relates the following terms.
- (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) State the effects of the following on the performance of a chromatographic column.
- (i) Raising the column temperature.
- (ii) Temperature programming. [7]
- (iii) Use illustrative diagrams to demonstrate the effects in (b) [3]

Question 2(25 marks)

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

(b) Distinguish 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 and define all the parameters in it. [3]
- (ii) Give two of the factors that influence the value of D. [2]

(d) A solute being extracted from water with carbon tetrachioride has a distribution ratio, D, of 85.0

(i) What percentage is extracted from the aqueous phase when 50.0mL of 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 [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) State 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) In 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) Calculate N and H for this column. [6]

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

(iii) Another peak next 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) Give three advantages of thin layer chromatography (TLC) over paper chromatography. [3]

(b) For the TLC.

(i) Give two examples each of the stationary phase and mobile phases commonly used for analysis. [4]

(ii) What stationary phases would you employ for:

- a polar compound
- a weakly polar compound [2]

(c) Briefly describe the procedure for chromatogram development and detection of analyte spots. [7]

(d) (i) Define 'R_f -Value' for the TLC. [1]

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

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

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	$R_\infty = m_e e^4/8h^3c\varepsilon_0^2$	$1.097\,37 \times 10^{5}$ 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}

PERIODIC TABLE OF ELEMENTS

GROUPS

PERIODS	1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8 VIIIB	9 VIIIB	10 VIIIB	11 IB	12 IIB	13 IIIA	14 IVA	15 VA	16 VIA	17 VIIA	18 VIIIA
1	1.008 H 1																	4.003 He 2
2	6.941 Li 3	9.012 Be 4											10.811 B 5	12.011 C 6	14.007 N 7	15.999 O 8	18.998 F 9	20.180 Ne 10
3	22.990 Na 11	24.305 Mg 12											26.982 Al 13	28.086 Si 14	30.974 P 15	32.06 S 16	35.453 Cl 17	39.948 Ar 18
4	39.098 K 19	40.078 Ca 20	44.956 Sc 21	47.88 Ti 22	50.942 V 23	51.996 Cr 24	54.938 Mn 25	55.847 Fe 26	58.933 Co 27	58.69 Ni 28	63.546 Cu 29	65.39 Zn 30	69.723 Ga 31	72.61 Ge 32	74.922 As 33	78.96 Se 34	79.904 Br 35	83.80 Kr 36
5	85.468 Rb 37	87.62 Sr 38	88.906 Y 39	91.224 Zr 40	92.906 Nb 41	95.94 Mo 42	98.907 Tc 43	101.07 Ru 44	102.91 Rh 45	106.42 Pd 46	107.87 Ag 47	112.41 Cd 48	114.82 In 49	118.71 Sn 50	121.75 Sb 51	127.60 Te 52	126.90 I 53	131.29 Xe 54
6	132.91 Cs 55	137.33 Ba 56	138.91 *La 57	178.49 Hf 72	180.95 Ta 73	183.85 W 74	186.21 Re 75	190.2 Os 76	192.22 Ir 77	195.08 Pt 78	196.97 Au 79	200.59 Hg 80	204.38 Tl 81	207.2 Pb 82	208.98 Bi 83	(209) Po 84	(210) At 85	(222) Rn 86
7	223 Fr 87	226.03 Ra 88	(227) **Ac 89	(261) Rf 104	(262) Ha 105	(263) Unh 106	(262) Uns 107	(265) Uno 108	(266) Une 109	(267) Uun 110								

TRANSITION ELEMENTS

Key: Atomic mass → 10.811; Symbol → B; Atomic No. → 5

Series														
*Lanthanide Series	140.12 Ce 58	140.91 Pr 59	144.24 Nd 60	(145) Pm 61	150.36 Sm 62	151.96 Eu 63	157.25 Gd 64	158.93 Tb 65	162.50 Dy 66	164.93 Ho 67	167.26 Er 68	168.93 Tm 69	173.04 Yb 70	174.97 Lu 71
**Actinide Series	232.04 Th 90	231.04 Pa 91	238.03 U 92	237.05 Np 93	(244) Pu 94	(243) Am 95	(247) Cm 96	(247) Bk 97	(251) Cf 98	(252) Es 99	(257) Fm 100	(258) Md 101	(259) No 102	(260) Lr 103

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