UNIVERSITY OF SWAZILAND

Faculty of Health Science

Department of Environmental Health Science

Main Semester I1 Examination

May 2012

Title of paper: Instrumental Methods for Environmental Analysis

Course code: EHS 574

Time allowed: 2 HOURS

Marks allocation: 100 Marks

Instructions:

1) ANSWER **ALL QUESTIONS**
2) Each question is weighted 25 marks
3) Write neatly and clearly
4) A periodic table and other useful data have been provided with this paper

DO NOT OPEN THIS QUESTION PAPER UNTIL PERMISSION TO DO SO HAS BEEN GRANTED BY THE CHIEF INVIGILATOR

Question 1 (25 marks)

(a) For the following sets of chromatographic terms, explain/define the terms in each set and give an expression that relates them.

(i) Rention time, t_R, adjusted retention time t'_R and dead time, t_M.

(ii) Capacity factor, k, retention time, t_R and dead time, t_M.

(iii) Volume flow rate, F, retention volume, V_R and retention time, t_R

(Each term needs to be defined only once). (9)

(b) Explain the terms resolution, R_s, between two adjacent peaks in a chromatogram. (2)

(c) A solute was eluted completely from a chromatographic column over a period of 2min. 24s. Calculate its retention volume if its flow rate is 24.0 mL/min. (4)

(d) During the chromatographic analysis of a sample, two adjacent peaks, A and B appear with the following features:

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

(i) Calculate the resolution between A and B. (3)

(ii) If the retention time for an unretained solute is 1.20 min, calculate the selectivity factor for A and B. (3)

(iii) Calculate the capacity factors for A and B. (4)

Question 2 (25 marks)

(a) (i) With the aid of an accompanying diagram, briefly describe the procedure for the analysis of a sample using the multiple point standard addition method. (8)

(ii) What is the main advantage of this method over the external standardization method? (2)

(b) During the flame emission spectroscopic determination of potassium, K in a given sample by the standard addition method, the following data were obtained:

Unknown (mL)	Added Standard (mL)	Final Volume (mL)	Emission Intensity (Arbitrary units)
5.00	0.00	50.00	309.0
5.00	2.50	50.00	380.5
5.00	5.00	50.00	454.5
5.00	7.50	50.00	537.0
5.00	10.00	50.00	607.5

If the concentration of the potassium standard used is 0.81 ppm-K. Calculate the concentration of potassium in the unknown. (15)

Question 3 (25 marks)

(a) Briefly describe the two major solvent extraction systems for metals. Give an example in each case. (5)

(b) Using an appropriate expression (without unnecessarily deriving it), show how the solvent extraction of metal chelates is affected by the pH of the system and the reagent (Ligand), concentration. (3)

(c) A compound, X, which weighs 1.200g is dissolved in 300 mL of water in a separatory funnel. If the K_D is 2.00, and assuming there is no dimerization and no pH dependence, calculate the amount of X that would remain after;

(i) One extraction with 300 mL of an organic solvent.
(ii) Three extractions with 100 mL of the organic solvent each time
(iii) Four extractions with 75 mL of the organic solvent each time.
(iv) Compare and comment on the results obtained in c(i-iii).

(17)

Question 4 (25 marks)

(a) Draw and label the schematic diagram of a Gas chromatograph (GC). (4)

(b) For the GC, briefly discuss:

(i) The requisite property and examples of the mobile phase. (2)
(ii) The main features of packed and open tubular columns. (6)
(iii) The advantages of open tubular columns over packed columns. (4)
(iv) The functions and ideal properties of the solid support and the stationary phase. (5)

(c) For the GC detector, discuss:

(i) Its function.
(ii) The factors determining its choice.
(iii) Its desirable properties.

(4)

PERIODIC TABLE OF ELEMENTS

GROUPS

PERIODS	1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8	9 VIIIB	10	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								

Atomic mass → 10.811
Symbol → B
Atomic No. → 5

TRANSITION ELEMENTS

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
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

*Lanthanide Series

**Actinide Series

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

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}