UNIVERSITY OF SWAZILAND

Faculty of Health Science

Department of Environmental Health Sciences

Final Examination 2014/15

Title of paper: Instrumental Methods for Environmental Analysis

Course Code: EHS 574

Time Allowed: 2 HOURS

Instructions:

1) Answer any Four (4) 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.

REQUIREMENT: GRAPH PAPER

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

Question 1 (25 marks)

(a) Given the following sets of terms, explain/define the terms in each set and give an expression that relates them.
(i) Retention 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) Determine 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) Estimate the capacity factors for A and B. (4)

Question 2 (25 marks)

(a) (i) Using an appropriate, supporting diagram, 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) The following data were obtained during the flame emission spectroscopic analysis of potassium, K in a given sample by the standard addition method.

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 0.81ppm-K, calculate the concentration of potassium in the unknown. (15)

Question 3 (25 marks)

(a) Discuss the basic principles of the two major solvent extraction systems for metals. Give an example in each case. (5)

(b) By employing the appropriate expression (without unnecessarily deriving it), describe the dependence of the solvent extraction of metal chelates on the pH of the system and the reagent (ligand), concentration. (3)

(c) A solute, X, which weighs 1.200g is dissolved in 300mL of water in a separatory funnel. If D = 2.00 for A and assuming there is no dimerization and no pH dependence, calculate the amount of X that would remain after;
(i) One extraction with 300mL of an organic solvent.
(ii) Three extractions with 100mL of the organic solvent each time
(iii) Four extractions with 75mL 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 a schematic diagram of the Gas chromatograph (GC). (4)

(b) For the GC, briefly discuss:
(i) The requisite property of a mobile phase. Give two examples. (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)

Question 5 (25 marks)

What is column efficiency with respect to gas chromatography (GC)? How is its value affected by N, the number of theoretical plates, and H, the plate height? What other factors influence it? (5)

(b) What is temperature programming in GC? Use a graphical illustration to show how it affects the resolution, R_s the retention time, t_R and the number of solutes eluted during a GC analysis. What are its advantages over the isothermal procedure? (11)

(c) Briefly discuss five general applications of 'Gas Chromatography'. Give four examples of industries and laboratories in Swaziland where this method is being used on routine basis. (9)

Question 6 (25 marks)

(a) Differentiate between 'Thin Layer Chromatography' (TLC) and 'Paper Chromatography', based on the following points of view:
   (i) The nature of the mobile phase.
   (ii) The nature of the stationary phase.
   (iii) Resolution and sensitivity. (6)

(b) Define R_f value, with regards to qualitative analysis in planar chromatography. (2)

(c) Employing the TLC method for the analysis of a polar substance summarize the procedure for the:
   (i) TLC plate preparation. (8)
   (ii) Identification of the separated components on the TLC plate. (9)

Periodic Table of the Elements

Atomic Number: 6
Symbol: C
Name: Carbon
Average Atomic Mass: 12.0107

Hydrogen
Semiconductors
(also known as metalloids)

Metals
Alkali metals
Alkaline earth metals
Transition metals
Other metals

Nonmetals
Halogens
Noble gases
Other nonmetals

	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11	Group 12	Group 13	Group 14	Group 15	Group 16	Group 17	Group 18
1	1 H Hydrogen 1.007 94																	2 He Helium 4.002 60
2	3 Li Lithium 6.941	[illegible]											5 B Boron 10.811	6 C Carbon 12.0107	7 N Nitrogen 14.0067	8 O Oxygen 15.9994	9 F Fluorine 18.998 4032	10 Ne Neon 20.1797
3	11 Na Sodium 22.989 769 28	[illegible]											13 Al Aluminum 26.981 5386	14 Si Silicon 28.0855	15 P Phosphorus 30.973 762	16 S Sulfur 32.065	17 Cl Chlorine 35.453	18 Ar Argon 39.948
4	19 K Potassium 39.0983	[illegible]	21 Sc Scandium 44.955 912	22 Ti Titanium 47.867	23 V Vanadium 50.9415	24 Cr Chromium 51.9961	25 Mn Manganese 54.938 045	26 Fe Iron 55.845	27 Co Cobalt 58.933 195	28 Ni Nickel 58.6934	29 Cu Copper 63.546	30 Zn Zinc 65.409	31 Ga Gallium 69.723	32 Ge Germanium 72.64	33 As Arsenic 74.921 60	34 Se Selenium 78.96	35 Br Bromine 79.904	36 Kr Krypton 83.798
5	37 Rb Rubidium 85.4678	[illegible]	39 Y Yttrium 88.905 85	40 Zr Zirconium 91.224	41 Nb Niobium 92.906 38	42 Mo Molybdenum 95.94	43 Tc Technetium (98)	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.905 50	46 Pd Palladium 106.42	47 Ag Silver 107.8682	48 Cd Cadmium 112.411	49 In Indium 114.818	50 Sn Tin 118.710	51 Sb Antimony 121.760	52 Te Tellurium 127.60	53 I Iodine 126.904 47	54 Xe Xenon 131.293
6	55 Cs Cesium 132.905 4519	[illegible]	57 La Lanthanum 138.905 47	72 Hf Hafnium 178.49	73 Ta Tantalum 180.947 88	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.084	79 Au Gold 196.966 569	80 Hg Mercury 200.59	81 Tl Thallium 204.3833	82 Pb Lead 207.2	83 Bi Bismuth 208.980 40	84 Po Polonium (209)	85 At Astatine (210)	86 Rn Radon (222)
7	87 Fr Francium (223)	[illegible]	89 Ac Actinium (227)	104 Rf Rutherfordium (261)	105 Db Dubnium (262)	106 Sg Seaborgium (266)	107 Bh Bohrium (264)	108 Hs Hassium (277)	109 Mt Meitnerium (268)	110 Ds Darmstadtium (271)	111 Rg Roentgenium (272)	112 Uub* Ununbium (285)		114 Uuq* Ununquadium (289)		116 Uuh* Ununhexium (292)		

* The systematic names and symbols for elements greater than 111 will be used until the approval of trivial names by the IUPAC.

The discoveries of elements with atomic numbers 112, 114, and 116 have been reported but not fully confirmed.

58 Ce Cerium 140.116	59 Pr Praseodymium 140.907 65	60 Nd Neodymium 144.242	61 Pm Promethium (145)	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.925 35	66 Dy Dysprosium 162.500	67 Ho Holmium 164.930 32	68 Er Erbium 167.259	69 Tm Thulium 168.934 21	70 Yb Ytterbium 173.04	71 Lu Lutetium 174.967
90 Th Thorium 232.038 06	91 Pa Protactinium 231.035 88	92 U Uranium 238.028 91	93 Np Neptunium (237)	94 Pu Plutonium (244)	95 Am Americium (243)	96 Cm Curium (247)	97 Bk Berkelium (247)	98 Cf Californium (251)	99 Es Einsteinium (252)	100 Fm Fermium (257)	101 Md Mendelevium (258)	102 No Nobelium (259)	103 Lr Lawrencium (262)

The atomic masses listed in this table reflect the precision of current measurements. (Each value listed in parentheses is the mass number of that radioactive element's most stable or most common isotope.)

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	$a_0 = 4\pi\varepsilon_0\hbar^2/m_e e$	$5.291\,77 \times 10^{-11}$ m
Rydberg constant	$R_\infty = m_e e^4/8h^3 c$	$1.097\,37 \times 10^{5}$ 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}