UNIVERSITY OF SWAZILAND
Faculty of Health Sciences
Department of Environmental Health Science

BSc IN ENVIRONMENTAL HEALTH SCIENCES

MAIN EXAMINATION PAPER 2017

TITLE OF PAPER : INSTRUMENTAL METHODS FOR
ENVIRONMENTAL ANALYSIS I

COURSE CODE : EHS209

DURATION : 2 HOURS

MARKS : 100

INSTRUCTIONS : READ THE QUESTIONS & INSTRUCTIONS
CAREFULLY

: ANSWER **ANY FOUR** QUESTIONS

: EACH QUESTION **CARRIES 25** MARKS.

: WRITE NEATLY & CLEARLY

: NO PAPER SHOULD BE BROUGHT INTO OR
OUT OF THE EXAMINATION ROOM.

: BEGIN EACH QUESTION ON A SEPARATE
SHEET OF PAPER.

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

QUESTION ONE

a. Identify whether the following statements are true or false. For each answer, give a reason why.

   (i)   Solid phase extraction technique can also be used as a concentration method.

   (ii)   In TLC, two components with the same RF value are identical.

   (iii) Random errors affect measurement accuracy.

   (iv)   Column efficiency is not affected by low oven temperatures in GC

   [4×4 Marks]

b. Using an example, explain why sample preparation is essential for environmental analysis? **[5 Marks]**

c. In chromatography, what is meant by retention factor/time?   **[4 Marks]**

QUESTION TWO

a. A TLC plate was developed using a 45 mL, 1:3:1 mixture of butanone, dichloromethane and hexane, respectively. Calculate the elution strength of this solution. (Required data is provided)   **[6 Marks]**

b. Describe the operating principles of an electron capture detector (ECD)

   [7 Marks]

c. Two TLC plates mounted with the same sample were developed using two different solvents. On TLC plate 1, no separation was seen up to the solvent front (all components were on the solvent front) while on TLC plate 2, all components were separated. Give reasons why:

   (i) There is no separation in TLC plate 1.

   (ii) The solvent used to develop TLC plate 1 is assumed to be non-polar.

   (iii) The solvent used to develop TLC plate 2 has an ideal elution strength

   [3 × 4 Marks]

QUESTION THREE

a. In a given TLC plate, what conclusion can be drawn for a component that has an RF value of;

   (i)   0.11

   (ii) 0.98

   [6 Marks]

b. Using equations discuss solvent extraction and include its disadvantages in the extraction of analytes from aqueous to organic phase.   **[8 Marks]**

c. Are multiple batch extractions a remedy for the disadvantages you have given in your answer for question b.?   **[4 Marks]**

d. In a chromatographic analysis of a mixture of chlorinated pesticides, in which a 2.0 m long column was used, a peak with retention time t_r, of 8.68 min and a baseline width of 0.36 min, was identified as dieldrin.

   (i)   Calculate N and H for this column   **[4 Marks]**

   (ii) Suggest a suitable detector for the analysis.   **[3 Marks]**

QUESTION FOUR

a. Lead levels were measured in Umzimnene river water using two instrumental methods. The samples were collected from two sampling points namely, Site A and Site B. Processed results are are shown in table 1.

Table 1: Lead analysis data for the two methods

	Potentiometry	Spectrophotometry
Site 1	0.69 ±0.005 ppm	0.63 ±0.008 ppm
	n=7	n=5
Site 2	0.78 ±0.007 ppm	0.87 ±0.008 ppm
	n=5	n=5

(i) What statistical test can be carried out to determine whether the two methods have similar standard deviations in both sites. With the data provided, is the statistical test possible? If not, justify.

[9 Marks]

(ii) For each method, what are confidence intervals for the measurements for Site 1? **[7 Marks]**

b. Discuss the Plate theory in gas chromatography (use diagrams and equations in your discussion). **[9 Marks]**

QUESTION FIVE

a. What are the advantages of microwave acid digestion over wet digestion?

[7 Marks]

a. The distribution constant of analyte X between n-Hexane and water is 8.9. Calculate the % E of the extraction of X after 50.0 mL of 0.200 M X is treated by extraction with one 20 mL portion of n-Hexane.

[12 Marks]

b. What is meant by the term "sample matrix effect"? How can this effect be corrected? **[6 Marks]**

Tabulated values for the Q-test

	0.822	0.941	0.970	0.988	0.994
	0.603	0.765	0.829	0.889	0.926
	0.488	0.642	0.710	0.780	0.821
	0.421	0.560	0.625	0.698	0.740
	0.375	0.507	0.568	0.637	0.680
	0.343	0.468	0.526	0.590	0.634
	0.319	0.437	0.493	0.555	0.598
	0.299	0.412	0.466	0.527	0.568
	0.271	0.375	0.425	0.480	0.518
	0.250	0.350	0.397	0.447	0.483
	0.234	0.329	0.376	0.422	0.460
	0.223	0.314	0.358	0.408	0.438
	0.213	0.300	0.343	0.392	0.420

Table 3.1

Values of t for v Degrees of Freedom for Various Confidence Levels[a]

v	Confidence Level			
	90%	95%	99%	99.5%
1	6.314	12.706	63.657	127.32
2	2.920	4.303	9.925	14.089
3	2.353	3.182	5.841	7.453
4	2.132	2.776	4.604	5.598
5	2.015	2.571	4.032	4.773
6	1.943	2.447	3.707	4.317
7	1.895	2.365	3.500	4.029
8	1.860	2.306	3.355	3.832
9	1.833	2.262	3.250	3.690
0	1.812	2.228	3.169	3.581
5	1.753	2.131	2.947	3.252
0	1.725	2.086	2.845	3.153
5	1.708	2.060	2.787	3.078
)	1.645	1.960	2.576	2.807

$= N - 1 =$ degrees of freedom.

Solvent	MF MW	Bp (°C) Density (g/mL)	Hazards*	Dipole	Elution Stength (ε)
Hexane $CH_3(CH_2)_4CH_3$	C_6H_{14} 86.17	68.7 0.659	Flammable Toxic	0.08	0.01
Toluene $C_6H_5CH_3$	C_7H_8 92.13	110.6 0.867	Flammable Toxic	0.31	0.22
Diethyl ether $CH_3CH_2OCH_2CH_3$	$C_4H_{10}O$ 74.12	34.6 0.713	Flammable Toxic, CNS Depressant	1.15	0.29
Dichloromethane CH_2Cl_2	CH_2Cl_2 84.94	39.8 1.326	Toxic, Irritant Cancer suspect	1.14	0.32
Ethyl Acetate $CH_3CO_2CH_2CH_3$	$C_4H_8O_2$ 88.10	77.1 0.901	Flammable Irritant	1.88	0.45
Acetone CH_3COCH_3	C_3H_6O 58.08	56.3 0.790	Flammable Irritant	2.69	0.43
Butanone $CH_3CH_2COCH_3$	C_4H_8O 72.10	80.1 0.805	Flammable Irritant	2.76	0.39
1-Butanol $CH_3CH_2CH_2CH_2OH$	$C_4H_{10}O$ 74.12	117.7 0.810	Flammable Irritant	1.75	0.47
Propanol $CH_3CH_2CH_2OH$	C_3H_8O 60.09	82.3 0.785	Flammable Irritant	1.66	0.63
Ethanol CH_3CH_2OH	C_2H_6O 46.07	78.5 0.789	Flammable Irritant	1.70	0.68
Methanol CH_3OH	CH_4O 32.04	64.7 0.791	Flammable Toxic	1.7	0.73
Water HOH	H_2O 18.02	100.0 0.998		1.87	>1

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	$F = N_A e$	9.6485×10^4 C mol^{-1}
Boltzmann constant	k	$1.380\ 66 \times 10^{-23}$ J K^{-1}
Gas constant	$R = N_A k$	$8.314\ 51$ J K^{-1} mol^{-1}
		$8.205\ 78 \times 10^{-2}$ dm^3 atm K^{-1} mol^{-1}
		6.2364×10 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		
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 permittivity	$\varepsilon_o = 1/c^2\mu_o$	$8.854\ 19 \times 10^{-12}$ J^{-1} C^2 m^{-1}
	$4\pi\varepsilon_o$	$1.112\ 65 \times 10^{-10}$ J^{-1} C^2 m^{-1}
Vacuum permeability	μ_o	$4\pi \times 10^{-7}$ J s^2 C^{-2} m^{-1}
		$4\pi \times 10^{-7}$ T^2 J^{-1} m^3
Magneton		
Bohr	$\mu_B = e\hbar/2m_e$	$9.274\ 02 \times 10^{-24}$ J T^{-1}
nuclear	$\mu_N = e\hbar/2m_p$	$5.050\ 79 \times 10^{-27}$ J T^{-1}
g value	g_e	$2.002\ 32$
Bohr radius	$a_o = 4\pi\varepsilon_o\hbar/m_e e^2$	$5.291\ 77 \times 10^{-11}$ m
Fine-structure constant	$\alpha = \mu_o e^2 c/2h$	$7.297\ 35 \times 10^{-3}$
Rydberg constant	$R_\infty = m_e e^4/8h^3 c\varepsilon_o^2$	$1.097\ 37 \times 10^7$ m^{-1}
Standard acceleration of free fall	g	$9.806\ 65$ m s^{-2}
Gravitational constant	G	$6.672\ 59 \times 10^{-11}$ N m^2 Kg^{-2}

Conversion factors

1 cal	=	4.184 joules (J)	1 erg	=	1×10^{-7} J
1 eV	=	$1.602\ 2 \times 10^{-19}$ J	1 eV/molecule	=	96 485 kJ mol^{-1}

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	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

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

*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	238.03 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.