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

DEGREE IN ENVIRONMENTAL HEALTH SCIENCES

RE-SIT EXAMINATION PAPER 2017

TITLE OF PAPER	: INSTRUMENTAL METHODS FOR ENVIRONMENTAL ANALYSIS I
COURSE CODE	: EHS 209
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) Standard deviation is used to describe the accuracy of a method.

   (ii) In chromatography, fronting and tailing affect peak resolution.

   (iii) The efficiency of solvent extraction (liquid-liquid) depends on K_D.

   (iv) Carrier gas flow rate does not affect the resolution of peaks in GC analysis.

[4×4 Marks]

b. In point form, outline the process multiple batch extraction using solvent extraction/ liquid-liquid extraction. **[5 Marks]**

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

QUESTION TWO

a. 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 origin) while on TLC plate 2, all components were separated. Give reasons why;

   (i) There is no separation in TLC plate 1. **[3 Marks]**

   (ii) The solvent used to develop TLC plate 1 is assumed to be very polar. **[5 Marks]**

   (iii) The solvent used to develop TLC plate 2 has ideal elution strength. **[3 Marks]**

b. For the given terms/phrases below, match each term to the relevant type of detector.

   (i) Heated filament

   (ii) Beta emitter

   (iii) Reduction of current

   (iv) Comparison of thermal conductivities

   (v) Electrophilic functional groups

(vi) Hydrogen/ air flame

(vii) Impact ionization

[2 × 7 Marks]

QUESTION THREE

a. What is 'column efficiency' in gas chromatography? **[5 Marks]**

b. How is column efficiency influenced by the following factors? (Use appropriate equations where necessary)

(i) 'loading' of the column,

(ii) N (number of theoretical plates) and

(iii) H (height of plate)? What other factors influence it? **[12 Marks]**

c. In a chromatographic analysis of a mixture of chlorinated pesticides, in which a 1.50 m long column was used, a peak with retention time t_r, of 8.68 min and a baseline width of 0.66 min, was identified as dieldrin.

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

(ii) Determine the capacity factor for dieldrin if the dead time, t_m, for the column is 0.30 Min. **[4 Marks]**

QUESTION FOUR

a. Explain what is an internal standard and how does it improve the precision of an instrumental measurement. **[8 Marks]**

b. Discuss the advantages of microwave acid digestion over wet digestion.

[5 Marks]

c. The gravimetric analysis of a Nickel compound was developed and compared to a spectrophotometric method. The w/w percentage of Nickel in the compound was reported in table 1.

Table 1: Analysis results from two methods

Gravimetric analysis (w/w%)	**Spectrophotometric analysis (w/w%)**
20.10	18.89
20.50	19.20
18.65	19.00
19.25	19.70
19.40	19.40
19.90	

(i) Is there significant difference between the standard deviations of the two methods? **[6 Marks]**

(ii) In each data set, are there outliers? Use the appropriate statistical test to reject data points. **[6 Marks]**

QUESTION FIVE

a. The distribution constant of analyte X between n-Hexane and water is 8.9. Calculate the concentration of of X remaining in the aqueous phase after 50.0 mL of 0.200 M X is treated by extraction with three 20 mL portions of n-Hexane. **[12 Marks]**

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

c. Draw a representative diagram of a typical GC instrument. **[7 Marks]**

Tabulated values for the Q-test

N	68%	90%	95%	98%	99%
3	0.822	0.941	0.970	0.988	0.994
4	0.603	0.765	0.829	0.889	0.926
5	0.488	0.642	0.710	0.780	0.821
6	0.421	0.560	0.625	0.698	0.740
7	0.375	0.507	0.568	0.637	0.680
8	0.343	0.468	0.526	0.590	0.634
9	0.319	0.437	0.493	0.555	0.598
10	0.299	0.412	0.466	0.527	0.568
12	0.271	0.375	0.425	0.480	0.518
14	0.250	0.350	0.397	0.447	0.483
16	0.234	0.329	0.376	0.422	0.460
18	0.223	0.314	0.358	0.408	0.438
20	0.213	0.300	0.343	0.392	0.420

Table 3.2

Values of F at the 95% Confidence Level

	$\nu_1 = 2$	3	4	5	6	7	8	9	10	15	20	30
$\nu_2 = 2$	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4	19.4	19.4	19.4	19.5
3	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81	8.79	8.70	8.66	8.62
4	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.86	5.80	5.75
5	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.62	4.56	4.50
6	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	3.94	3.87	3.81
7	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.64	3.51	3.44	3.38
8	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.35	3.22	3.15	3.08
9	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.01	2.94	2.86
10	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.85	2.77	2.70
15	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.40	2.33	2.25
20	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39	2.35	2.20	2.12	2.04
30	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21	2.16	2.01	1.93	1.84

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 X 10^{8} m s^{-1}
Elementary charge	e	1.602 177 X 10^{-19} C
Faraday constant	$F = N_A e$	9.6485 X 10^{4} C mol^{-1}
Boltzmann constant	k	1.380 66 X 10^{-23} J K^{-1}
Gas constant	$R = N_A k$	8.314 51 J K^{-1} mol^{-1}
		8.205 78 X 10^{-2} dm^{3} atm K^{-1} mol^{-1}
		6.2364 X 10 L Torr K^{-1} mol^{-1}
Planck constant	h	6.626 08 X 10^{-34} J s
	$\hbar = h/2\pi$	1.054 57 X-10^{-34} J s
Avogadro constant	N_A	6.022 14 X 10^{23} mol^{-1}
Atomic mass unit	u	1.660 54 X 10^{-27} Kg
Mass		
electron	m_e	9.109 39 X 10^{-31} Kg
proton	m_p	1.672 62 X 10^{-27} Kg
neutron	m_n	1.674 93 X 10^{-27} Kg
Vacuum permittivity	$\varepsilon_o = 1/c^2\mu_o$	8.854 19 X 10^{-12} J^{-1} C^{2} m^{-1}
	$4\pi\varepsilon_o$	1.112 65 X 10^{-10} J^{-1} C^{2} m^{-1}
Vacuum permeability	μ_o	4π X 10^{-7} J s^{2} C^{-2} m^{-1}
		4π X 10^{-7} T^{2} J^{-1} m^{3}
Magneton		
Bohr	$\mu_B = e\hbar/2m_e$	9.274 02 X 10^{-24} J T^{-1}
nuclear	$\mu_N = e\hbar/2m_p$	5.050 79 X 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 X 10^{-11} m
Fine-structure constant	$\alpha = \mu_o e^2 c/2h$	7.297 35 X 10^{-3}
Rydberg constant	$R_\infty = m_e e^4/8h^3c\varepsilon_o^2$	1.097 37 X 10^{7} m^{-1}
Standard acceleration of free fall	g	9.806 65 m s^{-2}
Gravitational constant	G	6.672 59 X 10^{-11} N m^{2} Kg^{-2}

Conversion factors

1 cal	=	4.184 joules (J)	1 erg	=	1 X 10^{-7} J
1 eV	=	1.602 2 X 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	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
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

*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.