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

DEGREE IN ENVIRONMENTAL HEALTH SCIENCES

MAIN EXAMINATION PAPER 2018

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. Match the terms on the left column with the suitable terms on the right.

	Column 1	**Column 2**
(i)	R_f value	a. GC
(ii)	Column efficiency	b. FID
(iii)	High quality blanks	c. Microwave acid digestion
(iv)	Concentration method	d. Poor resolution
(v)	R_t	e. SPME
(vi)	B emmitter	f. ECD
(vii)	Polymer coated fibre tip	g. Plate theory
(viii)	Wide peak base	h. Precision
(ix)	Air-Hydrogen flame	i. SPE
(x)	Standard deviation	j. TLC

[2 ×10 Marks]

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

QUESTION TWO

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

b. Identify whether the following statements are True or false. For each answer, give reasons 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.

    (v)   Random errors affect measurement accuracy.

[4×5 Marks]

QUESTION THREE

a. Two TLC plates mounted with the same sample was developed using two different solvents. On TLC plate A, no separation was seen from the origin (all components were on the origin) while on TLC plate B, all components were separated. Give reasons why;

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

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

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

[3 × 4 Marks]

b. 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.

[8 Marks]

c. What is meant by the term "sample matrix effect"? How can this effect be corrected? **[5 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]

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

c. Define or give a mathematical equation for the following terms:
    (i)   Precision
    (ii) Gaussian distribution
    (iii) Determinate error
    (iv) Primary standard

                                                                       [3 × 4 Marks]

QUESTION FIVE

a. Draw a schematic diagram of a typical gas chromatography instrument

                                                                       [8 Marks]

b. Are multiple batch extractions a solution for the disadvantages liquid-liquid extraction? Give reasons why.   [5 Marks]

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

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	$\varepsilon_0 = 1/c^2\mu_0$	$8.854\,19 \times 10^{-12}$ J^{-1} C^2 m^{-1}
	$4\pi\varepsilon_0$	$1.112\,65 \times 10^{-10}$ J^{-1} C^2 m^{-1}
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
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	ge	$2.002\,32$
Bohr radius	$a_0 = 4\pi\varepsilon_0\hbar/m_e e^2$	$5.291\,77 \times 10^{-11}$ m
Fine-structure constant	$\alpha = \mu_0 e^2 c/2h$	$7.297\,35 \times 10^{-3}$
Rydberg constant	$R_\infty = m_e e^4/8h^3 c\varepsilon_0^2$	$1.097\,37 \times 10^7$ 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	=	1.6022×10^{-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

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	(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.