

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

DEGREE IN ENVIRONMENTAL HEALTH SCIENCES

MAIN EXAMINATION PAPER 2019

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 <u>CARRIES 25</u> 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. Draw and label a schematic diagram of gas chromatography instrument.

[10 Marks]

- b. What are the properties of an ideal stationary phase of a GC column
 [9 Marks]
- c. Discuss the key assumptions necessary for the use of standards addition calibration method. [6 Marks]

Total 25 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
 - Microwave acid digestion is fast but results in a complex sample matrix.
 - (ii) In TLC, interactions between components and mobile phase depends polarities.
 - (iii) The efficiency of solvent extraction (liquid-liquid) of weak organic acids from aqueous phase depends on K_D .
 - (iv) Oven temperature does not affect the resolution of peaks in GC analysis.
 - (v) The student T-test compares means of different methods.

[4×5 Marks]

Total 25 marks

QUESTION THREE

a. Two TLC plates mounted with the same sample was developed using two different solvents. On TLC plate A, the 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 is assumed to be non-polar.
- (iii) The solvent used to develop TLC plate B has ideal elution strength

 $[3 \times 4 Marks]$

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.

[8 Marks]

b. Why is SPE also regarded as a method of concentrating a sample? [5 Marks]

Total 25 marks

QUESTION FOUR

- a. State sequentially, the steps that should be followed in solving a given analytical problem (i.e. in the analysis of a given sample).
 [10 Marks]
- b. Give reasons why calibration is an important part of all instrumental analysis procedures.
 [7 Marks]
- c. Name one method of calibration and outline the procedure for your chosen calibration method. [8 Marks]

Total: 25 marks

QUESTION FIVE

a. Discuss the differences between SPE and SPME.

[10 Marks]

- b. Are multiple batch extractions a solution for the disadvantages liquid-liquid extraction? Give reasons why. [5 Marks]
- c. In a chromatographic analysis of a mixture of chlorinated pesticides, in which a 2.0 m long column was used, three peaks were obtained. The peaks had the following characteristics;

Peak A: t_r = 2.01 min, W_b =0.094 min Peak B: t_r = 5.94 min, W_b = 0.782 min Peak C: t_r = 8.68 min, W_b = 0.36 min

- (i) Calculate the resolution between these peaks.
- [6 Marks]
- (ii) Use Peak B to calculate plate height, H and number of theoretical plates, N.

[4 Marks] Total 25 marks

PERIODIC TABLE OF ELEMENTS

	81	VIIIA	4,003	Hc	~	20.180	Ne	10	39.948	Ar	50	83.80	Ϋ́	36	131.29	X	54	(222)	R	86			
	17	VIIA		•	• .	18.998	Ľ,	6	35.453	ប	-2	79.904	Br	35	126.90	Н	53	(210)	At	85			
	91	VIA				15,999	0	60	32.06	S	91	78.96	Sc	34	127.60	ţ	-25	(204)	Po	84			
	15	Ϋ́				14.007	z	7	30.974	ፈ	15	74.922	As	33	121.75	Sb	51	208.98	Bi	. 83			
	14	Υ×				12.011	υ	9	28.086	Š	4	72.61	පී	32	118.71	Sin	50	207.2	Pb	82			
	13	IIIA				10.811	20 1	رم ا	26.982	¥	Ω.	69.723	5	31	114.82	In	49	204.38	F	81			
	12	113				Atomic mass —	Symbol -	Atomic No.				65.39	Zu	30	112.41	Ü	48	200.59	Hg	80			
	11	8				Atom	Syr	Aton		ENTS	63.546	<u>ವ</u>	53	107.87	Ag	47	196.97	Ψn	79				
	10										58.69	Z	28	106.42	Pd	46	195.08	꿃	78	(292)	Unn	≘	
GROUPS	6	VIIIB									58.933	රි	27	102.94	뙶	45	192.22	브	11	(200)	Une		
G	8			•						Y ELEM	55.847	2	26	101:07	Ru	44	190.2	ő	92	(202)	Ono	<u>8</u>	
	7	VIIB							TRANSITION ELEMENTS	54.938	Ψ		6	T ₂		186.21	Re	75	(292)	Uns	107		
	9	VIB						51.996		ပံ	24	95.94	Mo	42	183.85	3	74	(263)	Cul	8			
	5	٩B									50.942	>	23	92.906	ź	41	180.95	Ta	73	(292)	Ha	3	
	4	IVB									<u> </u>			91.224	Ķ	\$	178.49	Hf	72	(261)	Rf	<u>5</u>	
	3	1118			4						44.956	Sc	21	88.906	>-	39	138.91	*La	57	(227)	** Ac	68	
	2	ΥII				9.012	Be	4	24:305	_		40.078	ర్	20	87.62	ş	38	137,33	Ba	56	226.03	Ra	88
		<u></u> ≤	1.008	Ξ	-	6.941	ï	3	22.990	Ra	=	39.098	<u>~</u>	61	85.468	18	37	132.91	ű	55	223	Fr	87
		PERIODS		-			7			(1)	·		4			v		:	9			7	

20.12 S8	140.12 140.91 Ce Pr 58 59	14424 Nd 60	C P P P P P P P P P P P P P P P P P P P	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
232.04 231. Th Pa	231.04 Pa	238.03 U	237.05 Np	(244) Pu	(243) Am	(247) Cm	(247) Bk	(251) Cf	(252) Es	(257) Fm	(258) Md	(259) No	(260) Lr
8	16	26	93	ጀ	25	96	44	86	66	001	101	102	501
	() indi	cales the	e mass m	umber of	The isot	ope will.	the lon	test half	-life.				

. *Lanthanide Series

**Actinide Series

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 ⁻³³ 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
	$\hbar = \hbar/2\pi$	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_{ϵ}	9.109 39 X 10 ⁻³¹ Kg
proton	m_p	1.672 62 X 10 ⁻²⁷ Kg
neutron .	$\mathbf{m}_{\mathtt{s}}$	1.674 93 X 10 ⁻²⁷ Kg
Vacuum permittivity	$\varepsilon_{o} = I/c^{2}\mu_{o}$	8.854 19 X 10 ⁻¹² J ⁻¹ C ² m ⁻¹
	4πε,	1.112 65 X 10 ⁻¹⁰ J ⁻¹ C ² m ⁻¹
Vacuum permeability	μ_o	$4\pi \times 10^{-7} \text{ J s}^2 \text{ C}^{-2} \text{ m}^{-1}$
		$4\pi \times 10^{-7} \mathrm{T}^2 \mathrm{J}^1 \mathrm{m}^3$
Magneton		
Bohr	$\mu_{\rm B} = e\hbar/2m_{\rm e}$	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	8e	2.002 32
Bohr radius	$a_0 = 4\pi \epsilon_0 \hbar/m_e c^2$	5.291 77 X 10 ⁻¹¹ m
Fine-structure constant	$\alpha = \mu_0 e^2 c/2h$	⁻ 7.297 35 X 10 ⁻³
Rydberg constant	$R_{**} = m_e e^4 / 8h^3 c \epsilon_s^2$	1.097 37 X 10 ⁷ m ⁻¹
Standard acceleration	•	
of free fall	g	9.806 65 m s ⁻²
Gravitational constant	∗Ğ.	6.672 59 X 10 ⁻¹¹ N m ² Kg ⁻²

Conversion factors

1 cal = 1 eV =		joules (2 X 10		1 erg 1 eV/n	nolecul	e ·	342 252	1 X 1 96 48	0.7 J 5 kJ mo	[- ¹
Prefixes	femto		nano	μ micro 10 ⁻⁶	milli	centi		kilo	M mega 10 ⁶	G giga 10°