



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

DEGREE IN ENVIRONMENTAL HEALTH SCIENCES

RE-SIT EXAMINATION PAPER 2019

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. What is 'column efficiency' in gas chromatography? How is its value influenced by 'loading' of the column, N (number of theoretical plates) and H (height of plate)? What other factors influence it? **[8 Marks]**
- b. State the advantages and disadvantages of open tubular columns over packed columns used for GC analysis and outline the structural differences between these two columns **[7 Marks]**
- c. 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 **[6 Marks]**
- (ii) Determine the capacity factor for dieldrin if the dead time, t_m , for the column is 0.30 min. **[4 Marks]**

QUESTION TWO

- a. Distinguish between the following terms;
- (i) Precision and accuracy.
- (ii) Precision and bias. **[4 Marks]**
- b. Why is sample pre-treatment necessary before carrying out the actual analysis on a given sample? Give four examples of pre-treatment steps often employed in analytical laboratories. **[8 Marks]**
- c. Why should the chemical environment of a sample be properly controlled during analysis? Give one such control measure that could be taken to assure accuracy of obtained data. **[4 Marks]**

- d. State sequentially, the steps that should be followed in solving a given analytical problem (i.e. in the analysis of a given sample). **[9 Marks]**

QUESTION THREE

- a. Define the following terms;
- (i) Dynamic range
 - (ii) Detection limit
 - (iii) Systematic error
 - (iv) Sensitivity
 - (v) Outlier
- [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 standard addition calibration method. **[6 Marks]**

QUESTION FOUR

- a. Give three advantages of thin layer chromatography over paper chromatography. **[3 Marks]**
- b. For TLC;
- (i) Give two examples each of stationary phase and mobile phase **[4 Marks]**
 - (ii) What stationary phase would be used for a polar compound and a weakly polar compound? **[2 Marks]**
- c. Briefly describe the procedure for chromatogram development and detection of analyte spots in TLC. **[6 Marks]**
- d. Define R_f value for TLC. **[2 Marks]**
- e. Using a diagram, illustrate how the R_f value can be measured. **[4 Marks]**

- f. Give four factors that influence the R_f value of a compound [4 Marks]

QUESTION FIVE

- a. The distribution constant of analyte X between n-Hexane and water is 8.9. Calculate the concentration 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. Explain why the drying step is essential in sample preparation of biological samples for metal analysis. [4 Marks]
- d. In solvent extraction, what does a distribution coefficient of 1 mean? [3 Marks]

General data and fundamental constants

Quantity	Symbol	Value
Speed of light	c	$2.997\ 924\ 58 \times 10^8 \text{ m s}^{-1}$
Elementary charge	e	$1.602\ 177 \times 10^{-19} \text{ C}$
Faraday constant	$F = N_A e$	$9.6485 \times 10^4 \text{ C mol}^{-1}$
Boltzmann constant	k	$1.380\ 66 \times 10^{-23} \text{ J K}^{-1}$
Gas constant	$R = N_A k$	$8.314\ 51 \text{ J K}^{-1} \text{ mol}^{-1}$
		$8.205\ 78 \times 10^{-2} \text{ dm}^3 \text{ atm K}^{-1} \text{ mol}^{-1}$
		$6.2364 \times 10 \text{ L Torr K}^{-1} \text{ mol}^{-1}$
Planck constant	h	$6.626\ 08 \times 10^{-34} \text{ J s}$
	$\hbar = h/2\pi$	$1.054\ 57 \times 10^{-34} \text{ J s}$
Avogadro constant	N_A	$6.022\ 14 \times 10^{23} \text{ mol}^{-1}$
Atomic mass unit	u	$1.660\ 54 \times 10^{-27} \text{ Kg}$
Mass		
electron	m_e	$9.109\ 39 \times 10^{-31} \text{ Kg}$
proton	m_p	$1.672\ 62 \times 10^{-27} \text{ Kg}$
neutron	m_n	$1.674\ 93 \times 10^{-27} \text{ Kg}$
Vacuum permittivity	$\epsilon_0 = 1/c^2 \mu_0$	$8.854\ 19 \times 10^{-12} \text{ J}^{-1} \text{ C}^2 \text{ m}^{-1}$
	$4\pi\epsilon_0$	$1.112\ 65 \times 10^{-10} \text{ J}^{-1} \text{ C}^2 \text{ m}^{-1}$
Vacuum permeability	μ_0	$4\pi \times 10^{-7} \text{ J s}^2 \text{ C}^{-2} \text{ m}^{-1}$
		$4\pi \times 10^{-7} \text{ T}^2 \text{ J}^{-1} \text{ m}^3$
Magneton		
Bohr	$\mu_B = e\hbar/2m_e$	$9.274\ 02 \times 10^{-24} \text{ J T}^{-1}$
nuclear	$\mu_N = e\hbar/2m_p$	$5.050\ 79 \times 10^{-27} \text{ J T}^{-1}$
g value	g_e	2.002 32
Bohr radius	$a_0 = 4\pi\epsilon_0\hbar/m_e e^2$	$5.291\ 77 \times 10^{-11} \text{ m}$
Fine-structure constant	$\alpha = \mu_0 e^2 c/2h$	$7.297\ 35 \times 10^{-3}$
Rydberg constant	$R_\infty = m_e e^4/8h^3 c \epsilon_0^2$	$1.097\ 37 \times 10^7 \text{ m}^{-1}$
Standard acceleration of free fall	g	$9.806\ 65 \text{ m s}^{-2}$
Gravitational constant	G	$6.672\ 59 \times 10^{-11} \text{ N m}^2 \text{ Kg}^{-2}$

Conversion factors

1 cal	=	4.184 joules (J)	1 erg	=	$1 \times 10^{-7} \text{ J}$
1 eV	=	$1.602\ 2 \times 10^{-19} \text{ J}$	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	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	
	IA	IIA	IIIB	IVB	VB	VIB	VII B	VIII B	IX	X	IB	II B	IIIA	IVA	VA	VIA	VIIA	VIIIA	
1	H 1.008																		
2	Li 6.941	Be 9.012											B 10.811	C 12.011	N 14.007	O 15.999	F 18.998	Ne 20.180	
3	Na 22.990	Mg 24.305											Al 26.982	Si 28.086	P 30.974	S 32.06	Cl 35.453	Ar 39.948	
4	K 39.098	Ca 40.078	Sc 44.956	Ti 47.88	V 50.942	Cr 51.996	Mn 54.938	Fe 55.847	Co 58.933	Ni 58.69	Cu 63.546	Zn 65.39	Ga 69.723	Ge 72.61	As 74.922	Se 78.96	Br 79.904	Kr 83.80	
5	Rb 85.468	Sr 87.62	Y 88.906	Zr 91.224	Nb 92.906	Mo 95.94	Tc 98.907	Ru 101.07	Rh 102.91	Pd 106.42	Ag 107.87	Cd 112.41	In 114.82	Sn 118.71	Sb 121.75	Te 127.60	I 126.90	Xe 131.29	
6	Cs 132.91	Ba 137.33	*La 138.91	Hf 178.49	Ta 180.95	W 183.85	Re 186.21	Os 190.2	Ir 192.22	Pt 195.08	Au 196.97	Hg 200.59	Tl 204.38	Pb 207.2	Bi 208.98	Po (209)	At (210)	Rn (222)	
7	Fr 223	Ra 226.03	**Ac (227)	Rf (261)	Ha (262)	Unh (263)	Uns (262)	Uno (265)	Une (266)	Uun (267)									

TRANSITION ELEMENTS

140.12	140.91	144.24	150.36	151.96	157.25	158.93	162.50	164.93	167.26	168.93	173.04	174.97
Ce 58	Pr 59	Nd 60	Sm 62	Eu 63	Gd 64	Tb 65	Dy 66	Ho 67	Er 68	Tm 69	Yb 70	Lu 71
232.04	231.04	238.03	244	243	247	247	251	252	257	258	259	260
Th 90	Pa 91	U 92	Pu 94	Am 95	Cm 96	Bk 97	Cf 98	Es 99	Fm 100	Md 101	No 102	Lr 103

*Lanthanide Series

**Actinide Series

() indicates the mass number of the isotope with the longest half-life.