

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

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DEGREE IN ENVIRONMENTAL HEALTH SCIENCES

RE-SIT 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 **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. 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 solution 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]

Total 25 marks

QUESTION TWO

- 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. Briefly account for the difference [5 Marks]
- c. Give two structural differences between them, (columns in b.) [5 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 tr, 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) Determine the capacity factor for dieldrin if the dead time, tm, for the column is 0.30 Min. [3 Marks]

Total 25 marks

QUESTION THREE

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 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. [7 Marks]
- d. Define Rf value for TLC [1 Marks]
- e. Using a diagram, illustrate how the Rf value is measured. [4 Marks]
- f. Give four factors that influence the Rf value of a compound [4 Marks]

Total 25 marks

QUESTION FIVE

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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. c. What are the following terms used to describe in data evaluation;
 - (i) Absolute error
 - (ii) Relative standard deviation
 - (iii) Coefficient of variation

 $[3 \times 2 \text{ Marks}]$

- c. 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? [3 Marks]

Total 25 marks

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General data and fundamental constants

Quantity .	Symbol	Value
Speed of light	c	2.997 924 58 X 10 ⁸ m s ⁻¹
Elementary charge	е	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
	$\hbar = \hbar/2\pi$	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	$\mathrm{m_e}$	9.109 39 X 10 ³¹ Kg
proton	m_p	1.672 62 X 10 ⁻²⁷ Kg
neutron .	$\mathbf{m}_{_{\mathbf{i}}}$	1.674 93 X 10 ⁻²⁷ Kg
Vacuum permittivity	$\varepsilon_{\rm o} = 1/c^2 \mu_{\rm o}$	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}	$4\pi \times 10^{-7} \text{ J s}^2 \text{ C}^{-2} \text{ m}^{-1}$
		$4\pi \times 10^{-7} \text{T}^3 \text{J}^4 \text{m}^3$
Magneton		
Bohr	$\mu_{\rm B} = e\hbar/2m_{\rm e}$	9.274 02 X 10 ⁻²⁴ J T ⁻¹
nuclear	$\mu_N = e N/2m_p$	5.050 79 X 10 ⁻²⁷ J T ⁻¹
g value	8e	2.002 32
Bohr radius	$a_0 = 4\pi \epsilon_0 h/m_e c^2$	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	$R_{-} = m_e e^4/8h^3c\epsilon_e^2$	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
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Conversion factors

1 cal = 1 eV =	4.184 joules (J) 1.602 2 X 10 ⁻¹⁹ J			1 erg 1 eV/n	nolecule	· ·	=	134,10 0			
Prefixes	f p femto p 10 ⁻¹⁵ 1	oico	nano	micro	m - milli 10 ⁻³	centi			M mega 10 ⁶	G giga 10°	