

**UNIVERSITY OF SWAZILAND  
FACULTY OF HEALTH SCIENCES  
SUPPLEMENTARY EXAMINATION 2014/2015**

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**TITLE OF PAPER : Instrumental Methods For  
Environmental Analysis - 2**

**COURSE NUMBEER : EHS 574**

**TIME ALLOWED : Two(2) Hours**

**INSTRUCTIONS : Answer any four (4) questions. Each  
Question carries 25 marks.**

**A periodic table and other useful data have been provided with this paper.**

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**REQUIREMENT: GRAPH PAPER**

*You are not supposed to open this paper until permission to do so has been granted by the Chief Invigilator.*

**Question 1(25 marks)**

- (a) What is an internal standard? [1]
- (b) Describe the graphical method of the standard addition method in AAS/FES during quantitative elemental analysis. Give one unique advantage of this method. [7]
- (c) Why is an internal standard most appropriate for quantitative analysis when unavoidable losses of analytes are expected during sample preparation? [2]
- (c) Aliquots of the standard solution of an element X were mixed with an unknown sample containing X for AAS analysis. The standard solution contained 1.00mg of X per liter. The following absorbance readings were obtained:

Vol. of Unknown (mL)	Vol. of standard (mL)	Total Volume (mL)	Absorbance
10.00	0	100.00	0.163
10.00	1.00	100.00	0.240
10.00	2.00	100.00	0.319
10.00	3.00	100.00	0.402
10.00	4.00	100.00	0.478

- (i) Calculate the concentration (in mg/L) of added standard to each solution. [5]
- (ii) Using graphical method, determine the concentration of X in the unknown. [10]

**Question 2(25 marks)**

- a) What is solvent extraction? [1]
- b) Define K (the distribution coefficient) and D (the distribution ratio). State any difference/s between them. [4]
- c) Briefly describe the procedure for the extraction of a solute contained in a 50.0mL aqueous phase using 100.0mL carbon tetrachloride [7]
- d) Using an appropriate expression, identify the factors that influence the distribution ratio (D) of an acid that is monomeric in both aqueous and organic phases and whose anion does not penetrate the organic layer. [4]

- e) i) Ninety percent of a certain solute is extracted when equal volumes of aqueous and organic phases are used. What will be the percent extracted if the volume of the organic phase is doubled? [6]
- ii) If, instead of doubling the volume of the organic phase, the extraction is carried out twice using the same volume of the organic phase, which of the two procedure would you prefer and why? [3]

**Question 3(25 marks)**

- a) Define the following chromatographic terms:
- i) Retention, time  $t_R$
- ii) Retention volume,  $V_R$  [2]
- b) Using an illustrative chromatogram, discuss how chromatographic methods can be employed for both quantitative and qualitative analysis of a sample. [7]
- c) Draw and label the schematic diagram of a 'Gas Chromatograph' (GC) [4]
- d) For the GC, discuss :
- i) The main features of a packed column. [4]
- ii) The function and the ideal properties of the solid support for the column. [4]
- iii) The function and the ideal properties of the liquid phase for the column. [4]

**Question 4(25 marks)**

- a) In gas chromatography (GC) what is column efficiency? How is its value affected by  $N$ , the number of theoretical plates, and  $H$ , the plate height? What other factors affect it? [5]
- b) What is temperature programming in GC? Use a graphical illustration to show how it affects the resolution,  $R$  the retention time,  $t_r$  and the number of solutes eluted during a GC analysis. What are its advantages over the isothermal procedure? [11]
- c) Give five general applications of 'Gas Chromatographic analysis'. Give four examples of an industries and laboratories in Swaziland where this method is being used on routine basis. [9]

**Question 5(25 marks)**

- a) Distinguish between 'Thin Layer Chromatography' (TLC) and 'Paper Chromatography' from the following points of view:
- i) the nature of the phase.
  - ii) the nature of the stationary phase
  - iii) resolution and sensitivity. [6]
- b) Define  $R_f$  value, with regards to qualitative analysis in planar chromatography. [2]
- c) For the analysis of a polar substance using the TLC method, give a brief procedure for the:
- i) TLC plate preparation [8]
  - ii) Identification of the separated components on the TLC plate. [9]

Quantity	Symbol	Value	General data and fundamental constants
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	$F = eN_A$	$9.6485 \times 10^4 \text{ C mol}^{-1}$	
Boltzmann constant	$k$	$1.380\,66 \times 10^{-23} \text{ J K}^{-1}$	
Gas constant	$R = kN_A$	$8.314\,51 \text{ J K}^{-1} \text{ mol}^{-1}$ $8.205\,78 \times 10^{-2} \text{ dm}^3 \text{ atm K}^{-1} \text{ mol}^{-1}$ $62.364 \text{ L Torr K}^{-1} \text{ mol}^{-1}$	
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 of 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 permeability†	$\mu_0$	$4\pi \times 10^{-7} \text{ J s}^2 \text{ C}^{-2} \text{ m}^{-1}$ $4\pi \times 10^{-7} \text{ T}^2 \text{ J}^{-1} \text{ m}^3$	
Vacuum permittivity	$\epsilon_0 = 1/c^2 \mu_0$	$8.854\,19 \times 10^{-12} \text{ J}^{-1} \text{ C}^2 \text{ m}^{-1}$ $4\pi\epsilon_0$ $1.112\,65 \times 10^{-10} \text{ J}^{-1} \text{ C}^2 \text{ m}^{-1}$	
Bohr magneton	$\mu_B = e\hbar/2m_e$	$9.274\,02 \times 10^{-24} \text{ J T}^{-1}$	
Nuclear magneton	$\mu_N = e\hbar/2m_p$	$5.050\,79 \times 10^{-27} \text{ J T}^{-1}$	
Electron $g$ value	$g_e$	2.002 32	
Bohr radius	$a_0 = 4\pi\epsilon_0\hbar^2/m_e e^2$	$5.291\,77 \times 10^{-11} \text{ m}$	
Rydberg constant	$R_\infty = m_e e^4/8h^3 c$	$1.097\,37 \times 10^5 \text{ cm}^{-1}$	
Fine structure constant	$\alpha = \mu_0 e^2 c/2h$	$7.297\,35 \times 10^{-3}$	
Gravitational constant	$G$	$6.672\,59 \times 10^{-11} \text{ N m}^2 \text{ kg}^{-2}$	
Standard acceleration of free fall†	$g$	$9.806\,65 \text{ m s}^{-2}$	

† Exact (defined) values

f	p	n	$\mu$	m	c	d	k	M	G	Prefixes
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	GROUPS																		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	
	IA 1.008	IIA 9.012	IIIB	IVB	VB	VIB	VIB	VIB	VIII	IB	IIIB	IIIA	IVA	VA	VIA	VIIA	VIIIA 4.001	IIc 2	
1	H 1																		
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 89	Rf 104	Ha 105	Unh 106	Uns 107	Uno 108	Une 109	Uun 110									

Atomic mass →  
Symbol →  
Atomic No. →

## 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.