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UNIVERSITY OF SWAZILAND
SECOND SEMESTER EXAMINATION 2007/2008

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

COURSE NUMBER : **EHS 574**

TIME ALLOWED : **Three(3) 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.

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) For the following atomic spectroscopic methods, state whether it is emission or absorption, and give the analytical signal/quantity measured in each case.
- (i) FES, (ii) FAAS, (iii) EAAS. (iv) ICP. [4]
- (b) State the difference in the instrumental setup of FASS and FES. [2]
- (c) What is a 'hollow cathode lamp' in FASS? [2]
- (d) For the 'hollow cathode lamp' of FASS:
- (i) With the aid of a diagram in addition, describe its configuration. [6]
- (ii) Discuss the working principles. [8]
- (iii) What is the essence(importance), of the cup-like nature of the cathode? [1]
- (iv) What are the limitations of multi-element hollow cathode lamps? [2]

Question 2(25 marks)

- (a) For the EAAS technique :
- (i) With an additional illustrative diagram, describe the design of the electrothermal furnace. [5]
- (ii) Discuss the three main steps/processes involved in the atomization of an analyte in an electrothermal furnace. [7]
- (iii) What is the essence of using a matrix modifier during EAAS analysis? Give an example of a matrix modifier and the elemental analysis for which it is employed. [3]
- (iv) State two functions of the inert gas that flows through the furnace during an EAAS analysis.
- (v) Give four advantages and two disadvantages of EAAS over FAAS. [6]
- (b) In each of the following pairs of atomic spectroscopic methods, identify the one that has better sensitivity:
- (i) FES & AFS (ii) FAAS & EAAS
- (iii) FES & EAAS.

Question 3(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 graphical illustration to show how it affects the resolution, R_s , the retention time, t_r and the number of solutes eluded during a GC analysis. [5]
- (c) What are the functions and the ideal properties of the liquid stationary phase for GC analysis? [5]
- (d) Give four general applications of 'Gas Chromatographic analysis'. Give an example of an industry in Swaziland where this method is being used on routine bases. [5]

Question 4(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. [6]
- (c) Draw and label the schematic diagram of a 'Gas Chromatographic' (GC) [5]
- (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 ideal properties of the liquid for the column. [4]

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 mobile phase.
 - (ii) The nature of the stationary phase.
 - (iii) Resolution and sensitivity. [8]
- (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. [7]

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 $\hbar = h/2\pi$	$6.626\,08 \times 10^{-34} \text{ J s}$ $1.054\,57 \times 10^{-34} \text{ J s}$	
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†	μ_0	$4\pi \times 10^{-7} \text{ J s}^2 \text{ C}^{-2} \text{ m}^{-1}$ $4\pi \times 10^{-7} \text{ T}^2 \text{ J}^{-1} \text{ m}^2$	
Vacuum permittivity	$\epsilon_0 = 1/c^2\mu_0$ $4\pi\epsilon_0$	$8.854\,19 \times 10^{-12} \text{ J}^{-1} \text{ C}^2 \text{ m}^{-1}$ $1.112\,65 \times 10^{-10} \text{ J}^{-1} \text{ C}^2 \text{ m}^{-1}$	
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	$R_\infty = m_e e^4/8h^3c$	$1.097\,37 \times 10^5 \text{ cm}^{-1}$	
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	μ	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	IIIB	IVB	VB	VIB	VIIIB	VIIIB	VIIIB	VIIIB	IB	IIIB	IIIA	IVA	VA	VIA	VIIA	VIIIA 4.001
1	H 1																	He 2
2	Li 3 6.941	Be 4 9.012																Ne 10 20.180
3	Na 11 22.990	Mg 12 24.305																Ar 18 39.948
4	K 19 39.098	Ca 20 40.078	Sc 21 44.956	Ti 22 47.88	V 23 50.942	Cr 24 51.996	Mn 25 54.938	Fe 26 55.847	Co 27 58.933	Ni 28 58.69	Cu 29 63.546	Zn 30 65.39	Ga 31 69.723	Ge 32 72.61	As 33 74.922	Se 34 78.96	Br 35 79.904	Kr 36 83.80
5	Rb 37 85.468	Sr 38 87.62	Y 39 88.906	Zr 40 91.224	Nb 41 92.906	Mo 42 95.94	Tc 43 98.907	Ru 44 101.07	Rh 45 102.91	Pd 46 106.42	Ag 47 107.87	Cd 48 112.41	In 49 114.82	Sn 50 118.71	Sb 51 121.75	Te 52 127.60	I 53 126.90	Xe 54 131.29
6	Cs 55 132.91	Ba 56 137.33	*La 57 138.91	Hf 72 178.49	Ta 73 180.95	W 74 183.85	Re 75 186.21	Os 76 190.2	Ir 77 192.22	Pt 78 195.08	Au 79 196.97	Hg 80 200.59	Tl 81 204.38	Pb 82 207.2	Bi 83 208.98	Po 84 (209)	At 85 (210)	Rn (222)
7	Fr 87 223	Ra 88 226.03	**Ac 89 (227)	Rf 104 (261)	Ha 105 (262)	Uuh 106 (263)	Uus 107 (262)	Uuo 108 (265)	Uue 109 (266)	Uun 110 (267)								

TRANSITION ELEMENTS

Atomic mass →
Symbol ←
Atomic No. ←

B 5 10.811	C 6 12.011	N 7 14.007	O 8 15.999	F 9 18.998
Al 13 26.982	Si 14 28.086	P 15 30.974	S 16 32.06	Cl 17 35.453

*Lanthanide Series

**Actinide Series

140.12	140.91	144.24	(145)	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	Pm 61	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	237.05	(244)	(243)	(247)	(247)	(251)	(252)	(257)	(258)	(259)	(260)
Th 90	Pa 91	U 92	Np 93	Pu 94	Am 95	Cm 96	Bk 97	Cf 98	Es 99	Fm 100	Md 101	No 102	Lr 103

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

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