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

B.Sc. Degree in Environmental Health Science

SUPPLEMENTARY EXAMINATION PAPER JULY 2016

TITLE OF PAPER	:	INSTRUMENTAL METHODS FOR ENVIRONMENTAL ANALYSIS
COURSE CODE	:	EHS573
DURATION	:	2 HOURS
MARKS	:	100
INSTRUCTIONS	:	THERE ARE FIVE QUESTIONS IN THIS EXAM.
	:	ANSWER ANY FOUR OUT OF THE FIVE THE QUESTIONS
	:	EACH QUESTION CARRIES A MAXIMUM MARK OF 25%

Question 1

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)

b) (i) State the advantages and disadvantages of open tubular columns over packed columns used for GC analysis. Briefly account for the difference (5)

(ii) Give two structural differences between them (5)

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 (5)

(ii) Determine the capacity factor for dieldrin if the dead time, t_m, for the column is 0.30 Min. (2)

[25]

Question 2

a) Distinguish between the following terms;

(i) Precision and accuracy

(ii) Precision and bias (4)

b) What are the factors to consider before choosing an appropriate method for the analysis of a given sample? (4)

c) Why is sample pre-treatment necessary before carrying out the actual analysis on a give sample? Give four examples of pre-treatment steps often employed in analytical laboratories. (5)

d) 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. (3)

e) State sequentially, the steps that should be followed in solving a given analytical problem (i.e. in the analysis of a given sample). (5)

f) Define the detection limit of an analytical method. Using a labelled figure, illustrate the useful concentration range of an analytical method. (4)

[25]

Question 3

a) Give three advantages of thin layer chromatography over paper chromatography. (3)

b) For TLC;
  (i) Give two examples each of stationary phase and mobile phase (4)
  (ii) What stationary phase would be used for a polar compound and a weakly polar compound? (2)

c) Briefly describe the procedure for chromatogram development and detection of analyte spots in TLC. (7)

d) (i) Define R_f value for TLC (1)
  (ii)Using a diagram, illustrate how it can be measured. (4)

e) Give four factors that influence the R_f value of a compound (4)

[25]

Question 4

a) Draw and label a schematic diagram of gas chromatography instrument. (10)

b) For a GC detector, discuss;
  (i) Its function
  (ii) The factors determining its choice
  (iii) Its desirable properties

(9)

c) Discuss standard addition calibration the key assumptions are necessary to apply standards addition calibration. (6)

[25]

Question 5

The concentration of sulphur in a sample of diesel has been given as 50 ppm. However, when a chemist analysed the sulphur content of the sample 5 times, she obtained the following results; 43 ppm, 61 ppm, 52 ppm, 48 ppm, and 44 ppm.

(a) Calculate the average, standard deviation, coefficient of variation and standard error of the data set. (12)

(b) Calculate the 95% confidence interval. (4)

(c) If the accepted value for the concentration of sulphur in the sample is 50 ppm, are the results for this set of measurements significantly different at the 95% confidence level by the *t*-test. (5)

(d) Use the Q test to reject any outliers in the data set (4)

[25]

Appendix 1

Tabulated values for the Q-test

n	68%	90%	95%	98%	99%
3	0.822	0.941	0.970	0.988	0.994
4	0.603	0.765	0.829	0.889	0.926
5	0.488	0.642	0.710	0.780	0.821
6	0.421	0.560	0.625	0.698	0.740
7	0.375	0.507	0.568	0.637	0.680
8	0.343	0.468	0.526	0.590	0.634
9	0.319	0.437	0.493	0.555	0.598
10	0.299	0.412	0.466	0.527	0.568
12	0.271	0.375	0.425	0.480	0.518
14	0.250	0.350	0.397	0.447	0.483
16	0.234	0.329	0.376	0.422	0.460
18	0.223	0.314	0.358	0.408	0.438
20	0.213	0.300	0.343	0.392	0.420

Table 3.2

Values of *F* at the 95% Confidence Level

	$\nu_1 = 2$	3	4	5	6	7	8	9	10	15	20	30
$\nu_2 = 2$	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4	19.4	19.4	19.4	19.5
3	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81	8.79	8.70	8.66	8.62
4	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.86	5.80	5.75
5	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.62	4.56	4.50
6	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	3.94	3.87	3.81
7	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.64	3.51	3.44	3.38
8	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.35	3.22	3.15	3.08
9	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.01	2.94	2.86
10	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.85	2.77	2.70
15	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.40	2.33	2.25
20	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39	2.35	2.20	2.12	2.04
30	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21	2.16	2.01	1.93	1.84

Table 3.1

Values of t for ν Degrees of Freedom for Various Confidence Levels[a]

	Confidence Level			
ν	90%	95%	99%	99.5%
1	6.314	12.706	63.657	127.32
2	2.920	4.303	9.925	14.089
3	2.353	3.182	5.841	7.453
4	2.132	2.776	4.604	5.598
5	2.015	2.571	4.032	4.773
6	1.943	2.447	3.707	4.317
7	1.895	2.365	3.500	4.029
8	1.860	2.306	3.355	3.832
9	1.833	2.262	3.250	3.690
0	1.812	2.228	3.169	3.581
5	1.753	2.131	2.947	3.252
0	1.725	2.086	2.845	3.153
5	1.708	2.060	2.787	3.078
›	1.645	1.960	2.576	2.807

$= N - 1 =$ degrees of freedom.

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^{-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
	$\hbar = h/2\pi$	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_e	9.109 39 X 10^{-31} Kg
proton	m_p	1.672 62 X 10^{-27} Kg
neutron	m_n	1.674 93 X 10^{-27} Kg
Vacuum permittivity	$\varepsilon_o = 1/c^2\mu_o$	8.854 19 X 10^{-12} J^{-1} C^{2} m^{-1}
	$4\pi\varepsilon_o$	1.112 65 X 10^{-10} J^{-1} C^{2} m^{-1}
Vacuum permeability	μ_o	4π X 10^{-7} J s^{2} C^{-2} m^{-1}
		4π X 10^{-7} T^{2} J^{-1} m^{3}
Magneton		
Bohr	$\mu_B = e\hbar/2m_e$	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	g_e	2.002 32
Bohr radius	$a_o = 4\pi\varepsilon_o\hbar/m_e e^2$	5.291 77 X 10^{-11} m
Fine-structure constant	$\alpha = \mu_o e^2 c/2h$	7.297 35 X 10^{-3}
Rydberg constant	$R_\infty = m_e e^4/8h^3 c\varepsilon_o^{\,2}$	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}

Conversion factors

1 cal = 4.184 joules (J)

1 erg = 1 X 10^{-7} J

1 eV = 1.602 2 X 10^{-19} 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}

Periods	1A	2A	3B	4B	5B	6B	7B	8B			1B	2B	3A	4A	5A	6A	7A	8A
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)
1	1 H 1.008																	2 He 4.003
2	3 Li 6.941	4 Be 9.012											5 B 10.81	6 C 12.01	7 N 14.01	8 O 16.00	9 F 19.00	10 Ne 20.18
3	11 Na 22.99	12 Mg 24.31											13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95
4	19 K 39.10	20 Ca 40.08	21 Sc 44.96	22 Ti 47.87	23 V 50.94	24 Cr 52.00	25 Mn 54.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.69	29 Cu 63.55	30 Zn 65.41	31 Ga 69.72	32 Ge 72.64	33 As 74.92	34 Se 78.96	35 Br 79.90	36 Kr 83.80
5	37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.94	43 Tc [98]	44 Ru 101.07	45 Rh 102.91	46 Pd 106.42	47 Ag 107.87	48 Cd 112.41	49 In 114.82	50 Sn 118.71	51 Sb 121.76	52 Te 127.60	53 I 126.90	54 Xe 131.29
6	55 Cs 132.91	56 Ba 137.33	57 La 138.91	72 Hf 178.49	73 Ta 180.95	74 W 183.84	75 Re 186.21	76 Os 190.23	77 Ir 192.22	78 Pt 195.08	79 Au 196.97	80 Hg 200.59	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po [209]	85 At [210]	86 Rn [222]
7	87 Fr [223]	88 Ra [226]	89 Ac [227]	104 Rf [267]	105 Db [268]	106 Sg [271]	107 Bh [272]	108 Hs [270]	109 Mt [276]	110 Ds [281]	111 Rg [280]	112 Cn [285]	113 Uut [284]	114 Uuq [289]	115 Uup [288]	116 Uuh [293]	117 Uus [294]	118 Uuo [294]

Lanthanides	58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm [145]	62 Sm 150.36	63 Eu 151.96	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97
Actinides	90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np [237]	94 Pu [244]	95 Am [243]	96 Cm [247]	97 Bk [247]	98 Cf [251]	99 Es [252]	100 Fm [257]	101 Md [258]	102 No [259]	103 Lr [262]