

**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

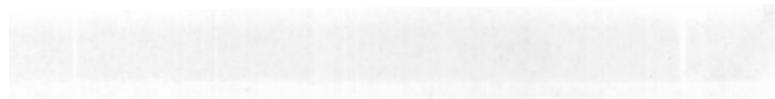
**COURSE CODE** : EHM204

**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%

EHM204  
SUPPLEMENTARY  
JULY 2016



### 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]

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### 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]

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### 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]**

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### 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]**

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**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)

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**[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

	$v_1 = 2$	3	4	5	6	7	8	9	10	15	20	30
$v_2 = 2$	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  $\nu$  Degrees of Freedom for Various Confidence Levels<sup>a</sup>**

$\nu$	Confidence Level			
	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
10	1.812	2.228	3.169	3.581
15	1.753	2.131	2.947	3.252
20	1.725	2.086	2.845	3.153
25	1.708	2.060	2.787	3.078
30	1.645	1.960	2.576	2.807

<sup>a</sup>  $N - 1 =$  degrees of freedom.

## 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	$\mu_0$	$4\pi \times 10^{-7} \text{ J s}^2 \text{ C}^{-2} \text{ m}^{-1}$ $4\pi \times 10^{-7} \text{ T}^2 \text{ J}^{-1} \text{ m}^3$
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	$a_0 = 4\pi\epsilon_0 \hbar^2 / m_e e^2$	$5.291\,77 \times 10^{-11} \text{ m}$
Fine-structure constant	$\alpha = \mu_0 e^2 c / 2\hbar$	$7.297\,35 \times 10^{-3}$
Rydberg constant	$R_\infty = m_e e^4 / 8\hbar^2 c \epsilon_0^2$	$1.097\,37 \times 10^7 \text{ m}^{-1}$
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	=	$96\,485 \text{ kJ mol}^{-1}$

Prefixes	f	p	n	$\mu$	m	c	d	k	M	G
	femto	pico	nano	micro	milli	centi	deci	kilo	mega	giga
	$10^{-15}$	$10^{-12}$	$10^{-9}$	$10^{-6}$	$10^{-3}$	$10^{-2}$	$10^{-1}$	$10^3$	$10^6$	$10^9$

1A (1)	2A (2)	8B						3A (13)	4A (14)	5A (15)	6A (16)	7A (17)	8A (18)				
1 H 1.008	2 Li 6.941	3 Na 22.99	4 K 39.10	5 Rb 85.47	6 Cs 132.91	7 Fr [223]	8 Be 9.012	9 Mg 24.31	10 Ca 40.08	11 Sr 87.62	12 Ba 137.33	13 Ra [226]	14 He 4.003				
							15 B 10.81	16 Al 26.98	17 Ga 69.72	18 In 114.82	19 Tl 204.38	20 Pb 207.2	21 Bi 208.98	22 Po [209]	23 At [210]	24 Rn [222]	
							25 C 12.01	26 Si 28.09	27 Ge 72.64	28 Sn 118.71	29 Pb 207.2	30 Bi 208.98	31 Uup [288]	32 Uuq [289]	33 Uuq [289]	34 Uuh [293]	35 Uuo [294]
							36 N 14.01	37 P 30.97	38 As 74.92	39 Sb 121.76	40 Bi 208.98	41 Uup [288]	42 Uuh [293]	43 Uuo [294]	44 Uuo [294]	45 Uuo [294]	46 Uuo [294]
							47 O 16.00	48 S 32.06	49 Se 78.96	50 Te 127.60	51 Po [209]	52 At [210]	53 Uuo [294]	54 Uuo [294]	55 Uuo [294]	56 Uuo [294]	57 Uuo [294]
							49 F 18.998	50 Cl 35.45	51 Br 79.90	52 I 126.90	53 At [210]	54 Uuo [294]	55 Uuo [294]	56 Uuo [294]	57 Uuo [294]	58 Uuo [294]	59 Uuo [294]
							51 Ne 20.18	52 Ar 39.95	53 Kr 83.80	54 Xe 131.29	55 Rn [222]	56 Uuo [294]	57 Uuo [294]	58 Uuo [294]	59 Uuo [294]	60 Uuo [294]	61 Uuo [294]
							53 He 4.003	54 Ne 20.18	55 Ar 39.95	56 Kr 83.80	57 Xe 131.29	58 Rn [222]	59 Uuo [294]	60 Uuo [294]	61 Uuo [294]	62 Uuo [294]	63 Uuo [294]
							55 H 1.008	56 Li 6.941	57 Na 22.99	58 K 39.10	59 Rb 85.47	60 Cs 132.91	61 Fr [223]	62 Uuo [294]	63 Uuo [294]	64 Uuo [294]	65 Uuo [294]
							57 Be 9.012	58 Mg 24.31	59 Ca 40.08	60 Sr 87.62	61 Ba 137.33	62 Ra [226]	63 Uuo [294]	64 Uuo [294]	65 Uuo [294]	66 Uuo [294]	67 Uuo [294]
							59 B 10.81	60 Al 26.98	61 Ga 69.72	62 In 114.82	63 Tl 204.38	64 Pb 207.2	65 Bi 208.98	66 Po [209]	67 At [210]	68 Rn [222]	69 Uuo [294]
							61 C 12.01	62 Si 28.09	63 Ge 72.64	64 Sn 118.71	65 Pb 207.2	66 Bi 208.98	67 Uuo [294]	68 Uuo [294]	69 Uuo [294]	70 Uuo [294]	71 Uuo [294]
							63 N 14.01	64 P 30.97	65 As 74.92	66 Sb 121.76	67 Bi 208.98	68 Uuo [294]	69 Uuo [294]	70 Uuo [294]	71 Uuo [294]	72 Uuo [294]	73 Uuo [294]
							65 O 16.00	66 S 32.06	67 Se 78.96	68 Te 127.60	69 Po [209]	70 At [210]	71 Uuo [294]	72 Uuo [294]	73 Uuo [294]	74 Uuo [294]	75 Uuo [294]
							67 F 18.998	68 Cl 35.45	69 Br 79.90	70 I 126.90	71 At [210]	72 Uuo [294]	73 Uuo [294]	74 Uuo [294]	75 Uuo [294]	76 Uuo [294]	77 Uuo [294]
							69 Ne 20.18	70 Ar 39.95	71 Kr 83.80	72 Xe 131.29	73 Rn [222]	74 Uuo [294]	75 Uuo [294]	76 Uuo [294]	77 Uuo [294]	78 Uuo [294]	79 Uuo [294]
							71 He 4.003	72 Ne 20.18	73 Ar 39.95	74 Kr 83.80	75 Xe 131.29	76 Rn [222]	77 Uuo [294]	78 Uuo [294]	79 Uuo [294]	80 Uuo [294]	81 Uuo [294]
							73 H 1.008	74 Li 6.941	75 Na 22.99	76 K 39.10	77 Rb 85.47	78 Cs 132.91	79 Fr [223]	80 Uuo [294]	81 Uuo [294]	82 Uuo [294]	83 Uuo [294]
							75 Be 9.012	76 Mg 24.31	77 Ca 40.08	78 Sr 87.62	79 Ba 137.33	80 Ra [226]	81 Uuo [294]	82 Uuo [294]	83 Uuo [294]	84 Uuo [294]	85 Uuo [294]
							77 B 10.81	78 Al 26.98	79 Ga 69.72	80 In 114.82	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po [209]	85 At [210]	86 Rn [222]	87 Uuo [294]
							79 C 12.01	80 Si 28.09	81 Ge 72.64	82 Sn 118.71	83 Pb 207.2	84 Bi 208.98	85 Uuo [294]	86 Uuo [294]	87 Uuo [294]	88 Uuo [294]	89 Uuo [294]
							81 N 14.01	82 P 30.97	83 As 74.92	84 Sb 121.76	85 Bi 208.98	86 Uuo [294]	87 Uuo [294]	88 Uuo [294]	89 Uuo [294]	90 Uuo [294]	91 Uuo [294]
							83 O 16.00	84 S 32.06	85 Se 78.96	86 Te 127.60	87 Po [209]	88 At [210]	89 Uuo [294]	90 Uuo [294]	91 Uuo [294]	92 Uuo [294]	93 Uuo [294]
							85 F 18.998	86 Cl 35.45	87 Br 79.90	88 I 126.90	89 At [210]	90 Uuo [294]	91 Uuo [294]	92 Uuo [294]	93 Uuo [294]	94 Uuo [294]	95 Uuo [294]
							87 Ne 20.18	88 Ar 39.95	89 Kr 83.80	90 Xe 131.29	91 Rn [222]	92 Uuo [294]	93 Uuo [294]	94 Uuo [294]	95 Uuo [294]	96 Uuo [294]	97 Uuo [294]
							89 He 4.003	90 Ne 20.18	91 Ar 39.95	92 Kr 83.80	93 Xe 131.29	94 Rn [222]	95 Uuo [294]	96 Uuo [294]	97 Uuo [294]	98 Uuo [294]	99 Uuo [294]
							91 H 1.008	92 Li 6.941	93 Na 22.99	94 K 39.10	95 Rb 85.47	96 Cs 132.91	97 Fr [223]	98 Uuo [294]	99 Uuo [294]	100 Uuo [294]	101 Uuo [294]
							93 Be 9.012	94 Mg 24.31	95 Ca 40.08	96 Sr 87.62	97 Ba 137.33	98 Ra [226]	99 Uuo [294]	100 Uuo [294]	101 Uuo [294]	102 Uuo [294]	103 Uuo [294]
							95 B 10.81	96 Al 26.98	97 Ga 69.72	98 In 114.82	99 Tl 204.38	100 Pb 207.2	101 Bi 208.98	102 Po [209]	103 At [210]	104 Rn [222]	105 Uuo [294]
							97 C 12.01	98 Si 28.09	99 Ge 72.64	100 Sn 118.71	101 Pb 207.2	102 Bi 208.98	103 Uuo [294]	104 Uuo [294]	105 Uuo [294]	106 Uuo [294]	107 Uuo [294]
							99 N 14.01	100 P 30.97	101 As 74.92	102 Sb 121.76	103 Bi 208.98	104 Uuo [294]	105 Uuo [294]	106 Uuo [294]	107 Uuo [294]	108 Uuo [294]	109 Uuo [294]
							101 O 16.00	102 S 32.06	103 Se 78.96	104 Te 127.60	105 Po [209]	106 At [210]	107 Uuo [294]	108 Uuo [294]	109 Uuo [294]	110 Uuo [294]	111 Uuo [294]
							103 F 18.998	104 Cl 35.45	105 Br 79.90	106 I 126.90	107 At [210]	108 Uuo [294]	109 Uuo [294]	110 Uuo [294]	111 Uuo [294]	112 Uuo [294]	113 Uuo [294]
							105 Ne 20.18	106 Ar 39.95	107 Kr 83.80	108 Xe 131.29	109 Rn [222]	110 Uuo [294]	111 Uuo [294]	112 Uuo [294]	113 Uuo [294]	114 Uuo [294]	115 Uuo [294]
							107 He 4.003	108 Ne 20.18	109 Ar 39.95	110 Kr 83.80	111 Xe 131.29	112 Rn [222]	113 Uuo [294]	114 Uuo [294]	115 Uuo [294]	116 Uuo [294]	117 Uuo [294]
							109 H 1.008	110 Li 6.941	111 Na 22.99	112 K 39.10	113 Rb 85.47	114 Cs 132.91	115 Fr [223]	116 Uuo [294]	117 Uuo [294]	118 Uuo [294]	119 Uuo [294]
							111 Be 9.012	112 Mg 24.31	113 Ca 40.08	114 Sr 87.62	115 Ba 137.33	116 Ra [226]	117 Uuo [294]	118 Uuo [294]	119 Uuo [294]	120 Uuo [294]	121 Uuo [294]
							113 B 10.81	114 Al 26.98	115 Ga 69.72	116 In 114.82	117 Tl 204.38	118 Pb 207.2	119 Bi 208.98	120 Po [209]	121 At [210]	122 Rn [222]	123 Uuo [294]
							115 C 12.01	116 Si 28.09	117 Ge 72.64	118 Sn 118.71	119 Pb 207.2	120 Bi 208.98	121 Uuo [294]	122 Uuo [294]	123 Uuo [294]	124 Uuo [294]	125 Uuo [294]
							117 N 14.01	118 P 30.97	119 As 74.92	120 Sb 121.76	121 Bi 208.98	122 Uuo [294]	123 Uuo [294]	124 Uuo [294]	125 Uuo [294]	126 Uuo [294]	127 Uuo [294]
							119 O 16.00	120 S 32.06	121 Se 78.96	122 Te 127.60	123 Po [209]	124 At [210]	125 Uuo [294]	126 Uuo [294]	127 Uuo [294]	128 Uuo [294]	129 Uuo [294]
							121 F 18.998	122 Cl 35.45	123 Br 79.90	124 I 126.90	125 At [210]	126 Uuo [294]	127 Uuo [294]	128 Uuo [294]	129 Uuo [294]	130 Uuo [294]	131 Uuo [294]
							123 Ne 20.18	124 Ar 39.95	125 Kr 83.80	126 Xe 131.29	127 Rn [222]	128 Uuo [294]	129 Uuo [294]	130 Uuo [294]	131 Uuo [294]	132 Uuo [294]	133 Uuo [294]
							125 He 4.003	126 Ne 20.18	127 Ar 39.95	128 Kr 83.80	129 Xe 131.29	130 Rn [222]	131 Uuo [294]	132 Uuo [294]	133 Uuo [294]	134 Uuo [294]	135 Uuo [294]
							127 H 1.008	128 Li 6.941	129 Na 22.99	130 K 39.10	131 Rb 85.47	132 Cs 132.91	133 Fr [223]	134 Uuo [294]	135 Uuo [294]	136 Uuo [294]	137 Uuo [294]
							129 Be 9.012	130 Mg 24.31	131 Ca 40.08	132 Sr 87.62	133 Ba 137.33	134 Ra [226]	135 Uuo [294]	136 Uuo [294]	137 Uuo [294]	138 Uuo [294]	139 Uuo [294]
							131 B 10.81	132 Al 26.98	133 Ga 69.72	134 In 114.82	135 Tl 204.38	136 Pb 207.2	137 Bi 208.98	138 Po [209]	139 At [210]	140 Rn [222]	141 Uuo [294]
							133 C 12.01	134 Si 28.09	135 Ge 72.64	136 Sn 118.71	137 Pb 207.2	138 Bi 208.98	139 Uuo [294]	140 Uuo [294]	141 Uuo [294]	142 Uuo [294]	143 Uuo [294]
							135 N 14.01	136 P 30.97	137 As 74.92	138 Sb 121.76	139 Bi 208.98	140 Uuo [294]	141 Uuo [294]	142 Uuo [294]	143 Uuo [294]	144 Uuo [294]	145 Uuo [294]
							137 O 16.00	138 S 32.06	139 Se 78.96	140 Te 127.60	141 Po [209]	142 At [210]	143 Uuo [294]				