



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

DEGREE IN ENVIRONMENTAL HEALTH SCIENCES

MAIN EXAMINATION PAPER 2016

TITLE OF PAPER : INSTRUMENTAL METHODS FOR ENVIRONMENTAL ANALYSIS I

COURSE CODE : EHM 204

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. Define the following terms;
- (i) Dynamic range
 - (ii) Detection limit
 - (iii) Bias
 - (iv) Systematic error
 - (v) Sensitivity
 - (vi) Outlier
- [2 × 6 Marks]**
- b. What sample preparation steps are involved in the analysis of metallic analytes in biological samples. **[8 Marks]**
- c. In chromatography, what is meant by retention factor? **[5 Marks]**

QUESTION TWO

- a. Explain what is an internal standard and how does it improve the precision of an instrumental measurement. **[8 Marks]**
- b. Discuss solvent extraction and include its disadvantages in the extraction of organics from water samples. **[8 Marks]**
- c. Explain how solid-phase micro-extraction (SPME) works and relate it to the principles of adsorption. **[9 Marks]**

QUESTION THREE

- a. Differentiate between a flame ionization detector (FID), a thermal conductivity detector (TCD) and an electron capture detector (ECD). **[12 Marks]**
- b. 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.
- (i) Calculate the average, standard deviation, coefficient of variation and standard error of the data set. **[6 Marks]**

(ii) Calculate the 95% confidence interval. **[3 Marks]**

(iii) Use the Q test to reject any outliers in the data set **[4 Marks]**

QUESTION FOUR

- a. Discuss the Plate theory in gas chromatography (use diagrams and equations in your discussion). **[10 Marks]**
- b. A TLC plate was developed using a 25 mL, 2:3 mixture of methanol and hexane, respectively. Calculate the elution strength of this solution. (Required data is provided) **[8 Marks]**
- c. Explain why such a mixture as the one in (b) would be used as opposed to using pure solvents. **[7 Marks]**

QUESTION FIVE

- a. What is 'column efficiency' in gas chromatography? **[5 Marks]**
- b. How is column efficiency influenced by the following factors? (Use appropriate equations where necessary)
- (i) 'loading' of the column,
 - (ii) N (number of theoretical plates) and
 - (iii) H (height of plate)? What other factors influence it? **[12 Marks]**
- 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 **[4 Marks]**
 - (iv) Determine the capacity factor for dieldrin if the dead time, t_m , for the column is 0.30 Min. **[4 Marks]**

Solvent	MF MW	Bp (°C) Density (g/mL)	Hazards*	Dipole	Elution Strength (ε)
Hexane CH ₃ (CH ₂) ₄ CH ₃	C ₆ H ₁₄ 86.17	68.7 0.659	Flammable Toxic	0.08	0.01
Toluene C ₆ H ₅ CH ₃	C ₇ H ₈ 92.13	110.6 0.867	Flammable Toxic	0.31	0.22
Diethyl ether CH ₃ CH ₂ OCH ₂ CH ₃	C ₄ H ₁₀ O 74.12	34.6 0.713	Flammable Toxic, CNS Depressant	1.15	0.29
Dichloromethane CH ₂ Cl ₂	CH ₂ Cl ₂ 84.94	39.8 1.326	Toxic, Irritant Cancer suspect	1.14	0.32
Ethyl Acetate CH ₃ CO ₂ CH ₂ CH ₃	C ₄ H ₈ O ₂ 88.10	77.1 0.901	Flammable Irritant	1.88	0.45
Acetone CH ₃ COCH ₃	C ₃ H ₆ O 58.08	56.3 0.790	Flammable Irritant	2.69	0.43
Butanone CH ₃ CH ₂ COCH ₃	C ₄ H ₈ O 72.10	80.1 0.805	Flammable Irritant	2.76	0.39
1-Butanol CH ₃ CH ₂ CH ₂ CH ₂ OH	C ₄ H ₁₀ O 74.12	117.7 0.810	Flammable Irritant	1.75	0.47
Propanol CH ₃ CH ₂ CH ₂ OH	C ₃ H ₈ O 60.09	82.3 0.785	Flammable Irritant	1.66	0.63
Ethanol CH ₃ CH ₂ OH	C ₂ H ₆ O 46.07	78.5 0.789	Flammable Irritant	1.70	0.68
Methanol CH ₃ OH	CH ₄ O 32.04	64.7 0.791	Flammable Toxic	1.7	0.73
Water HOH	H ₂ O 18.02	100.0 0.998		1.87	>1

APPENDIX

VALUES OF t FOR VARIOUS LEVELS OF PROBABILITY					
Number of Observations	Factor for Confidence Interval				
	80%	90%	95%	99%	99.90%
1	3.08	6.31	12.7	63.7	637
2	1.89	2.92	4.3	9.92	31.6
3	1.64	2.35	3.18	5.84	12.9
4	1.53	2.13	2.78	4.6	8.6
5	1.48	2.02	2.57	4.03	6.86
6	1.44	1.94	2.45	3.71	5.96
7	1.42	1.9	2.36	3.5	5.4
8	1.4	1.86	2.31	3.36	5.04
9	1.38	1.83	2.26	3.25	4.78
10	1.37	1.81	2.23	3.17	4.59
11	1.36	1.8	2.2	3.11	4.44
12	1.36	1.78	2.18	3.06	4.32
13	1.35	1.77	2.16	3.01	4.22
14	1.34	1.76	2.14	2.98	4.14

CRITICAL VALUES FOR REJECTION QUOTIENT Q					
Number of Observations	90%	95%	99%		
	Confidence	Confidence	Confidence		
3	0.941	0.970	0.994		
4	0.765	0.829	0.926		
5	0.642	0.710	0.821		
6	0.560	0.625	0.740		
7	0.507	0.568	0.680		
8	0.468	0.526	0.634		
9	0.437	0.493	0.598		
10	0.412	0.466	0.568		