



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 : EHS 209

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]

**APPENDIX**

VALUES OF <i>t</i> 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		

UNIVERSITY OF SWAZILAND  
Department of Chemistry

1	<b>H</b> 1.0079	2	<b>He</b> 4.0026	Atomic Number																Atomic Weight																																																																																																																																																																																					
3	<b>Li</b> 6.941	4	<b>Be</b> 9.0122	5	<b>B</b> 10.811	6	<b>C</b> 12.011	7	<b>N</b> 14.007	8	<b>O</b> 15.999	9	<b>F</b> 18.998	10	<b>Ne</b> 20.179	11	<b>Na</b> 22.990	12	<b>Mg</b> 24.305	13	<b>Al</b> 26.982	14	<b>Si</b> 28.086	15	<b>P</b> 30.974	16	<b>S</b> 32.064	17	<b>Cl</b> 35.453	18	<b>Ar</b> 39.948	19	<b>K</b> 39.098	20	<b>Ca</b> 40.078	21	<b>Sc</b> 44.956	22	<b>Ti</b> 47.88	23	<b>V</b> 50.942	24	<b>Cr</b> 51.996	25	<b>Mn</b> 54.938	26	<b>Fe</b> 55.847	27	<b>Co</b> 58.933	28	<b>Ni</b> 58.69	29	<b>Cu</b> 63.546	30	<b>Zn</b> 65.39	31	<b>Ga</b> 69.723	32	<b>Ge</b> 72.61	33	<b>As</b> 74.922	34	<b>Se</b> 78.96	35	<b>Br</b> 79.904	36	<b>Kr</b> 83.80	37	<b>Rb</b> 85.47	38	<b>Sr</b> 87.62	39	<b>Y</b> 88.906	40	<b>Zr</b> 91.224	41	<b>Nb</b> 92.906	42	<b>Mo</b> 95.94	43	<b>Tc</b> (98)	44	<b>Ru</b> 101.07	45	<b>Rh</b> 102.91	46	<b>Pd</b> 106.42	47	<b>Ag</b> 107.87	48	<b>Cd</b> 112.41	49	<b>In</b> 114.82	50	<b>Sn</b> 118.71	51	<b>Sb</b> 121.75	52	<b>Te</b> 127.60	53	<b>I</b> 126.90	54	<b>Xe</b> 131.29	55	<b>Cs</b> 132.91	56	<b>Ba</b> 137.33	57	<b>La</b> 138.91	58	<b>Ce</b> 140.12	59	<b>Pr</b> 140.91	60	<b>Nd</b> 144.24	61	<b>Pm</b> 146.92	62	<b>Sm</b> 150.36	63	<b>Eu</b> 151.97	64	<b>Gd</b> 157.25	65	<b>Tb</b> 158.93	66	<b>Dy</b> 162.50	67	<b>Ho</b> 164.93	68	<b>Er</b> 167.26	69	<b>Tm</b> 168.93	70	<b>Yb</b> 173.04	71	<b>Lu</b> 174.97	72	<b>Hf</b> 178.49	73	<b>Ta</b> 180.95	74	<b>W</b> 183.85	75	<b>Re</b> 186.2	76	<b>Os</b> 190.2	77	<b>Ir</b> 192.22	78	<b>Pt</b> 195.08	79	<b>Au</b> 196.97	80	<b>Hg</b> 200.59	81	<b>Tl</b> 204.38	82	<b>Pb</b> 207.2	83	<b>Bi</b> 208.98	84	<b>Po</b> (209)	85	<b>At</b> (210)	86	<b>Rn</b> (222)	87	<b>Fr</b> (223)	88	<b>Ra</b> 226.03	89	<b>Ac</b> 227.03	90	<b>Th</b> 232.04	91	<b>Pa</b> 231.04	92	<b>U</b> 238.03	93	<b>Np</b> 237.05	94	<b>Pu</b> (244)	95	<b>Am</b> (243)	96	<b>Cm</b> (247)	97	<b>Bk</b> 247	98	<b>Cf</b> (251)	99	<b>Es</b> (252)	100	<b>Fm</b> (257)	101	<b>Md</b> (258)	102	<b>No</b> (259)	103	<b>Lr</b> (260)