#### **DEPARTMENT OF CHEMISTRY**

#### **UNIVERSITY OF SWAZILAND**

#### **MAY 2015 FINAL EXAMINATION**

**TITLE OF PAPER** 

INTRODUCTION TO ANALYTICAL CHEMISTRY

**COURSE NUMBER** 

C204

TIME

: 3 HOURS

**Important Information** 

: 1. Each question is worth 25 marks.

2. Answer any four (4) questions in this paper.

3. Marks for ALL procedural calculations will be awarded.

4. Start each question on a fresh page of the answer sheet.

5. Diagrams must be large and clearly labelled accordingly.

6. This paper contains an appendix of chemical constants

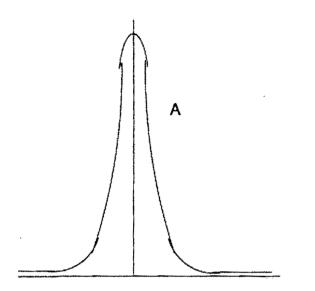
7. Additional material: graph paper.

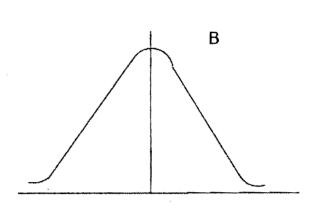
You are not supposed to open this paper until permission has been granted by the chief invigilator

- a) Define or explain the following:
  - i) Accuracy
  - ii) Determinate error
  - iii) Precision
  - iv) Indeterminate error
- (4)
- b) i) List the four main types of determinate error. (2)
- ii) Give a brief explanation/description of each of the types of determinate error you listed in
- (i). (4)
- iii) Explain two ways which can be used to detect determinate errors. (4)
- c) The following data were obtained for the analysis of a standard soil sample containing the analyte acenaphthene, a known carcinogen. The concentration of acenaphthene in the sample is given as 3.2 ppb

<u>Determination</u>	Acenaphthene (ppb)
1	3.0
2	2.9
3	3.1

- i) Calculate the mean of the data set.
- ii) Calculate the relative error of the mean of the data set.
- iii) Calculate the standard deviation of the small data set.
- iv) Calculate the confidence interval of the data set at a 95% confidence level.
- v) Explain in your own words what the confidence interval calculated in (vi) means. (8)
- d) The following curves A and B represent two data sets for an analyte in the same sample analyzed by two different analysts;





Which data set is the more precise A or B? Explain (3)

#### **QUESTION 2**

- a) Explain what is meant by standardization in titration and give one example of a primary standard used in acid-base titration to standardize HCl and one to standardize NaOH. (4)
- b) In the determination of chlorine by Fajan's titration in samples,
  - i) Name the common adsorption indicator used in this titration. (2)
  - ii) What is the reason for the addition of dextrin before titration? (2)
- c) You need to produce 500 mL of an aqueous solution in which the concentration of Pb is 10 ppm. Your source of Pb ions is lead (II) nitrate. How much lead (II) nitrate do you need to weigh out to produce this solution? (3)

d)

- i) Find the pH in the titration of 25 mL of 0.3 M HF with 0.3 M NaOH after adding 10 mL of NaOH. (5)
- ii) What name is given to the resultant solution in b (i)? (2)
- e) The standard hydrogen electrode (SHE) is the electrode against which all electrode potentials are referenced.

- i) Draw the SHE and label all components. What is the role for the platinum? Why is it a suitable metal for this role? (3)
- ii) What specifications should be met by the SHE? (2)
- iii) State the function of the salt bridge and explain how it works. (2)

- a) A young scientist determined the amount of Riboflavin (Vitamin B 2) in a cereal sample by measuring its fluorescence intensity in 5% acetic acid solution. A calibration curve was prepared by measuring the fluorescence intensities of a series of standards of increasing concentrations. The concentration of the standard was found to be 10.0 ppm. Explain in detail <u>TWO</u> ways which the young scientists can use to show that the method and instrument used for measurements gives accurate results. (Give a detailed explanation how this would be achieved) (5)
- b) An enzymatic method for determining alcohol in wine is evaluated by comparison with gas-chromatographic (GC) method. The sample is analysed several times by both methods with the following results (% ethanol).

Enzymatic Method: 13.1, 12.7, 12.6, 13.3, 13.3

GC Method: 13.5, 13.3, 13.0, 12.9.

Does the enzymatic method give the same value as the GC method at the 95 % confidence level? (5)

- c) One of the challenges in the quantification of elements is the problem of interferences. Explain what is meant by interferences giving a specific example and a solution on how this interference can be eliminated in analytical chemistry. (4)
- d) A standard method for the determination of carbon monoxide (CO) level in gaseous mixtures is known from many hundred measurements to have a standard deviation of 0.21 ppm CO. A modification of the method yields a value for s of 0.15ppm CO pooled from data set with 12 degrees of freedom. Does the modification give more precise measurements than the original method? (5)

- e) An atomic absorption method for the determination of copper content in fuels yielded a pooled standard deviation of spooled =  $0.32 \mu g \text{ Cu/mL (s} \rightarrow \sigma)$ . The analysis of the oil from a reciprocating aircraft engine showed a copper content of  $8.53 \mu g \text{ Cu/mL}$ .
  - i) Calculate the 99% confidence limits for the result based on a mean of four (4) analyses. (3)
  - ii) How many replicate measurements are necessary to decrease the 99% confidence for the analysis to  $\pm$  0.20 µg Cu/mL? (3)

a) The amount of iron (Fe) in drinking water was determined using the standard addition method. The concentration of the Fe standard was 11.1 ppm. The dilutions and signals for the different dilutions are given in the table below. Calculate the amount of Fe in the water sample in ppb.

Sample Volume	Standard Volume	
(mL)	(mL)	Signal (V)
10	0	0.215
10	5	0.424
10	_10	0.685
10	15	0.826
10	20	0.967

Note: All solutions were diluted to a final volume of 50 mL. (7)

b) External calibration is one other method which can be used in the determination of iron in water. Explain in detail how external calibration differs from standard addition and also give the main advantage of using standard addition over external calibration.

(5)

- c) Explain two disadvantages of using the standard addition method (3)
- d) Internal calibration is another method of calibration which is used in determination of the concentrations of unknown. In detail, explain the principle of internal standard, explain the advantage of using this type of method over the external calibration and standard addition. (7)
- e) Give the three (3) properties which an internal standard should meet. (3)

a) The CO in a 20.3 L sample of gas was converted to CO<sub>2</sub> by passing the gas over iodine pentoxide heated to 150 degrees Celsius.

$$I_2O_5(s) + 5CO(g) \rightarrow 5CO_2(g) + I_2(g)$$

The iodine was distilled at this temperature and was collected in an absorber containing 8.25 mL of 0.01101 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>

$$I_2(g) + 2S_2O_3^{2-}(aq) \rightarrow 2I^{-}(aq) + S_4O_6^{2-}(aq)$$

The excess Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> was back titrated with 2.16 mL of 0.00947 M I<sub>2</sub> solution.

- i) Calculate the concentration in milligrams of CO (28.01 g/mol) per litre of sample.
  (5)
- ii) The method used in a(i) is known as back titration, explain what is meant by back titration? (2)
- iii) Give four (4) purposes of back titration i.e. cases which would require the use of back titration instead of direct titration. (4)
- b) In titrimetry;
  - Differentiate between primary standard and a secondary standard for titrimetric analysis (2)
  - ii) Give four (4) desirable properties for a primary standard used for titration purposes. (4)
- c) Titration Curves describe how pH changes during various acid-base titrations. Draw the titration curve for the titration of 25 mL HCl of 0.100 M with 0.100M HCl. (5)

d) Using sketch diagrams, explain what would be the difference of a titration curve obtained when 10 mL 0.1 M acetic acid is titrated with 0.1 M sodium hydroxide from that of 0.1 M hydrochloric acid titrated with 0.1 M sodium hydroxide. (**Do not Calculate**) (3)

#### **QUESTION 6**

- a) One of the properties of a homogeneous precipitate in gravimetry is that it has fewer impurities. Describe the <u>two</u> (2) ways which can be used in the formation of a homogeneous precipitate. Give an example for each. (4)
- b) Explain how the particle size of a precipitate can be controlled with reference to relative supersaturation. (3)
- c) i) What is meant by 'digestion of a precipitate'? Briefly describe what happens in the process of digesting a precipitate and give <u>two</u> (2) advantages of this step during gravimetric analysis. (3)
  - ii) What is peptization? How can this phenomenon be avoided during gravimetric analysis (2)
- d) One of the gravimetric titration methods used in analytical chemistry is the Volhard titration.
  - i) Describe the Volhard titration method. What is this type of titration used for?(3)
  - ii) Give the two main challenges encountered when using this method of titration.(2)
  - iii) How can these problems listed in b(ii) be solved? (2)
- e) A 0.2356g sample containing only NaCl and BaCl2 Yielded 0.4637 g of dried Ag Cl on adding AgNO<sub>3</sub> to the sample. Calculate the percent of each halogen compound in the sample. (6)

### <u>APPENDIX</u>

VALUES OF £ FOR VARIOUS LEVELS OF PROBABILITY											
Number of Observations	Factor fo	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 ͺ	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 VAL	JES FOR REJE	CTION QUOTI	ENT Q	
Number of	90%	95%	99%	
Observations	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	7
9	0.437	0.493	0.598	
10	0.412	0.466	0.568	

# **Confidence Levels for Various Values** of z

-	
Confidence Level, %	z
	·
50	0.67
68	1.00
89	1.28
<b>S</b>	
90 /	1.64
·	
95	1.96
95.4	2.00
99	2.58
99.7	3.00
99.9	3.29

Table 4-5	Critical	values e	KF.	at K	%, conf	idence l	evel							
Degrees of						Degre	ees of fr	eedom f	or s <sub>1</sub>					
freedom													ŧ	,
for s <sub>2</sub>	2	3	4	5	6	7	8	9	10	12	15	20	30	<del>,</del> <del></del>
2	19.0	19.2	19.2	19.3	19.3	19,4	19.4	19,4	19,4	19.4	19,4	19.4	19.5	19.5
3	9.55	9.28	9.12	9,01	8.94	3.89	R.R4	3.81	8.79	8.74	8.70	8.66	8.62	8.53
4	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.91	5.86	5.80	5.75	5.63
5	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.63	4.62	4.56	4.50	4.36
4 5 6	5,14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	4.00	3.94	3.87	3.81	3.67
- 2	4,74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3,64	3.58	3.51	3,44	3.38	3.23
8	4.46	4,07	3,84	3.69	3.58	3.50	3.44	3.39	3.35	3.28	3.22	3,15	3.08	2.93
9	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.07	3.01	2.94	2.86	2.71
10	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.91	2.84	2.77	2.70	2.54
П	3.98	3.59	3.36	3.20	3.10	3.01	2.95	2.90	2.85	2.79	2.72	2.65	2.57	2.40
12	3.88	3,49	3.26	3.11	3.00	2.91	2.85	2.80	275	269	2.62	2.54	2.47	2.30
13	3.81	3,41	3 18	3.02	2 92	2.83	277	271	2.67	2.60	2,53	2.46	2.38	221
14	3,74	3.34	5.3.11	2.96	2.85	2.76	2.70	2.65	2.60	2.53	2.46	2.39	2.31	213
15	3.68	5.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.43	2.40	2.53	2.25	2.07
16	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.54	2.49	2.42	2,35	2.28	2.19	2.01
17	3.59	3.20	2.96	2.81	2.70	2.61	2.55	2.49	2.45	2.38	2.31	2.23	2.15	1.96
18	3.56	3.16	2.93	2.77	2.66	2.58	2.51	2.46	2.41	2.34	2,27	2.19	2.11	1.92
19	3.52	3.13	2.90	2.74	2.63	2.54	2.48	2.42	2.38	2.31	2.23	2.16	2.07	1.88
20	3.49	3.10	2.87	2.71	. 2.60	2.51	2.45	2.39	2.35	2.28	2,20	2.12	2.04	1.84
30	3.32	2.92	2.69	2:53	2.42	2.33	2.27	2.21	2.16	2.09	2.01	1.93	1.84	1.62
-7x	3.00	2.60	2.37	2,21	2.10	2,01	1.94	1,88	1,83	1.75	1,67	1.57	1.46	1.00

## **USEFUL CONSTANTS**

 $K_w = 1.00 \times 10^{-14}$ 

2

Ka	Acid		Base				
	Name	Formula	Formula	Name			
Large	Perchloric acid	HClO₄	ClO <sub>4</sub>	Perchlorate ion			
3.2 * 10 <sup>9</sup>	Hydroiodic acid	HI	I-	Iodide			
1.0 * 10 <sup>9</sup>	Hydrobromic acid	HBr	Br-	Bromide			
1.3 * 10 <sup>6</sup>	Hydrochloric acid	HCl	Cl-	Chloride			
$1.0 * 10^3$	Sulfuric acid	H <sub>2</sub> SO <sub>4</sub>	HSO <sub>4</sub>	Hydrogen sulfate ion			
$2.4 * 10^{1}$	Nitric acid	HNO <sub>3</sub>	NO <sub>3</sub>	Nitrate ion			
	Hydronium ion	H <sub>3</sub> O+	H <sub>2</sub> O	Water			

Ka 🐾	Acid		Conj	ugate Base		
*	Name	Formula	Formula	Name		
Large	Perchloric acid	HClO₄	ClO <sub>4</sub>	Perchlorate ion		
3.2 * 10 <sup>9</sup>	Hydroiodic acid	HI	I-	Iodide		
1.0 * 10 <sup>9</sup>	Hydrobromic acid	HBr	Br-	Bromide		
1.3 * 10 <sup>6</sup>	Hydrochloric acid	HCl	Cl-	Chloride		
$1.0 * 10^3$	Sulfuric acid	$H_2SO_4$	HSO <sub>4</sub> -	Hydrogen sulfate ion		
$2.4 * 10^{1}$	Nitric acid	$HNO_3$	NO <sub>3</sub>	Nitrate ion		
	Hydronium ion	H <sub>3</sub> O+	H <sub>2</sub> O	Water		
5.4 * 10 <sup>-2</sup>	Oxalic acid	$HO_2C_2O_2H$	$HO_2C_2O_2$	Hydrogen oxalate ion		
1.3 * 10-2	Sulfurous acid	$H_2SO_3$	HSO₃ -	Hydrogen sulfite ion		
1.0 * 10 <sup>-2</sup>	Hydrogen sulfate ion	HSO₄	SO <sub>4</sub> <sup>2-</sup>	Sulfate ion		
7.1 * 10 <sup>-3</sup>	Phosphoric acid	$H_3PO_4$	H <sub>2</sub> PO <sub>4</sub>	Dihydrogen phosphate ion		
7.2 * 10 <sup>-4</sup>	Nitrous acid	HNO <sub>2</sub>	NO <sub>3</sub>	Nitrite ion		
6.6 * 10 <sup>-4</sup>	Hydrofluoric acid	HF	F -	Fluoride ion		
1.8 * 10 <sup>-4</sup>	Methanoic acid	HCO <sub>2</sub> H	HCO <sub>2</sub>	Methanoate ion		
6.3 * 10 <sup>-5</sup>	Benzoic acid	C <sub>6</sub> H <sub>5</sub> COOH	C <sub>6</sub> H <sub>5</sub> COO-	Benzoate ion		
5.4 * 10 <sup>-5</sup>	Hydrogen oxalate ion	$HO_2C_2O^2$	$O_2C_2O_2^{2-}$	Oxalate ion		
1.8 * 10-5	Ethanoic acid	CH₃COOH	CH₃COO	Ethanoate (acetate)		
4.4 * 10 <sup>-7</sup>	Carbonic acid	CO <sub>3</sub> <sup>2-</sup>	HCO <sub>3</sub>	Hydrogen carbonate ion		
$\frac{1.1 * 10^{-7}}{6.3 * 10^{-8}}$	Hydrosulfuric acid	H <sub>2</sub> S	HS-	Hydrogen sulfide ion		
6.3 * 10 <sup>-8</sup>	Dihydrogen phosphate ion	H <sub>2</sub> PO <sub>4</sub>	HPO <sub>4</sub> <sup>2-</sup>	Hydrogen phosphate ion		
6.2 * 10-8	Hydrogen sulfite ion	HS-	S <sup>2-</sup>	Sulfite ion		
2.9 * 10-8	. Hypochlorous acid	ĤCIO	ClO <sup>-</sup>	Hypochlorite ion		
6.2 * 10 <sup>-10</sup>	Hydrocyanic acid	HCN	CN	Cyanide ion		
5.8 * 10 <sup>-10</sup> 5.8 * 10 <sup>-10</sup>	Ammonium ion	NH <sub>4</sub> <sup>+</sup>	NH <sub>3</sub>	Ammonia		
5.8 * 10 <sup>-10</sup>	Boric acid	H <sub>3</sub> BO <sub>3</sub>	H <sub>2</sub> BO <sub>3</sub>	Dihydrogen carbonate ion		
4.7 * 10 <sup>-11</sup>	Hydrogen carbonate ion	HCO <sub>3</sub> ·	CO <sub>3</sub> 2-	Carbonate ion		
4.2 * 10 <sup>-13</sup>	Hydrogen phosphate ion	HCO₃ - HPO₄ <sup>2-</sup>	PO <sub>4</sub> 3-	Phosphate ion		
1.8 * 10 <sup>-13</sup>	Dihydrogen borate ion	H <sub>2</sub> BO <sub>3</sub>	HBO <sub>3</sub> <sup>2-</sup>	Hydrogen borate ion		
1.3 * 10 <sup>-13</sup>	Hydrogen sulfide ion	HS-	S 2-	Sulfide ion		
1.6 * 10 <sup>-14</sup>	Hydrogen borate ion	HBO <sub>3</sub> <sup>2-</sup>	BO <sub>3</sub> 3-	Borate ion		
	water	H <sub>2</sub> O	OH-	Hydroxide		

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f	۶	n	μ	m		d	k	м	G	Prefixes	
femto	pico	nano	micro	milli	centi	deci	kilo	mega	olos		
10-15	10~13	10-	10-4	10-2		10-1		106			

## PERIODIC TABLE OF ELEMENTS

GROUPS	
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		2	3	4	5	6	7	8	9.*	10	11.	12.	13	14.	15	16 -		18
PERIODS	IA	IIA.	1110	IVII	VB	Vili	Viii	1	VIIIN		113	IIB	IIIA	IVA	٧٨	VIA	VIIA	Ailiv
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2	Li	Be											₽ B	C	N	0	F	Nc
1	3	4	1			•					Aton	iic No. ¯	7-5	6	7	8	9	10
	22.990	24.305	]		<b>*</b>								26.982	28,086	30.974	32.06	35.433	39.948
3	Nn	Mg	ľ			TRAN	SITIO	v elen	IENTS				Al	Si	P	S	CI	Ar
1.	11	12				•			•	•			13	14	15	16	17	18
	39.098	-40.078	44.956	47.88	50.942	51.996	54.938	55.847	58.933	58.69	63.546	65.39 .	69.723	72.61	74.922	78.96	79.904	83.80
4	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
	19	20	21	22	23	24	25	26	27	28	29	30.	31	32	33	34	35	36
	85.468	87.62	88.906	91.224	92.906	95.94	98.907	101.07	102.91	106.42	107.87	112.41	114.82	118.71	121.75	127.60	126.90	131.29
5	Rb	Šr	Y	Zr	"Nb"	Mô	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	1	Xe
	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54
	132.91	137.33	138.91	178.49	180.95	183.85	186.21	190.2	192,22	195.08	196.97	200.59	204.38	207.2	208.98	(209)	(210)	(222)
6		· Par	- La		r. The	r yaWagan	····Re····	· · · • • • • • • • • • • • • • • • • •	renan <b>ka</b> r	Pt	Au	Hg	Ti	PЬ	Bi	Po	Λt	fen
	- 55-	-36-	57	72	73		75.	76		78	79	80	81	82	83	84	85	86
	221	226.03	(227)	(261)	(267)	(201)	(262)	(263)	(266)	(267)							:	
7	Fr	Ra	**Ac	Rf	Ha	Unh	Uns	Uno	Une	Uun								
1	67 1	CONTRACT T	RO T	~ Yna~ l	*** 10° * 1	- 106	107	TOR I	tog l	110								

\*Lanthanide Series

\* † Actinitle 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	Pr	Nd	Pm	Sm	<u>R</u> u	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
58	59	60	61	62	63	64	65	66	67	68	69	70	71
90	231.04 Pa 91	92	93	(244) Pu 94	(243) Am. 25	(247) Cm 96	(247) .Bk 97	(251) Cf 98	(252) Es 99	(257) Fm 100	(258) Md 101	(259) No 102	(260) Lr 103

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