UNIVERSITY OF RUHUNA

BACHELOR OF SCIENCE HONORS IN MARINE AND FRESH WATER SCIENCES / BACHELOR OF SCIENCE IN FISHERIES AND MARINE SCIENCES DEGREE

LEVEL I SEMESTER I EXAMINATIONS - AUGUST/ SEPTEMBER -2018

CHM1122 Analytical Chemistry I

Answer **three (3)** questions only **Time: 01½** hours

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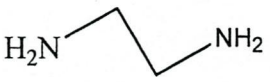
(01) Answer **all** parts.

(a) (i) Consider the following two reactions.

$M(H_2O)_6^{2+} + NH_3 \longrightarrow M(H_2O)_5NH_3^{2+} + H_2O$ $\log\beta = 12.6$

$M(H_2O)_6^{2+} + en \longrightarrow M(H_2O)_4(en)^{2+} + 2\,H_2O$ $\log\beta = 20.6$

Explain the trend in logβ values for the two metal complexes.

Note: en = 

(20 marks)

(ii) Briefly discuss the importance of EDTA as a complexing agent in titrations.

(10 marks)

(b) Explain under what conditions you would perform following titrations. Briefly give the titration procedures.

(i) Back titrations

(ii) Displacement titrations

(30 marks)

(c) 25.00 mL of M^{2+} ion solution was treated with excess amount of Mg-EDTA complex at pH 10 in the presence of EBT indicator. Titration of the resultant mixture consumed 18.55 mL of 0.01 M EDTA.

(i) Write the balanced chemical equations for the above titration.

(ii) Calculate the concentration of M^{2+} in the solution.

(iii) What is the requirement for the above titration to be quantitative ? (40 marks)

(02) Answer **all** parts.

(a)

(i) Briefly discuss the advantages and disadvantages of cerium over permanganate in redox titrations.

(15 marks)

(ii) Explain how you would minimize errors in iodometric titrations.

(15 marks)

(b) Potassium permanganate is used in wastewater treatments. Concentration of a permanganate solution is determined by reacting it with $Na_2C_2O_4$. A student dissolved 0.1322 g of $Na_2C_2O_4$ (FW 134 g/mol) in 100 mL distilled water. The content was acidified with H_2SO_4 acid and titrated with a $KMnO_4$ solution.

(i) Write down balanced chemical equations for the reactions between $C_2O_4^{2-}$ and MnO_4^-

(ii) Explain how you would determine the end point of the titration.

(iii) If the $Na_2C_2O_4$ solution consumed 22.50 mL of $KMnO_4$, calculate the concentration of $KMnO_4$ solution.

(35 marks)

(c)

(i) Defining the terms, write down the expression for the relative supersaturation pertaining to gravimetry.

(ii) Using the above expression, explain how you would maximize the crystal growth of a compound.

(35 marks)

(03) Answer **all** parts.

(a) Explain the following statements.

(i) NaOH cannot be used as a primary standard.

(ii) End point and equivalence point are not the same.

(20 marks)

(b) You are given 0.90 M CH_3COOH and 0.49 M $CH_3COO^-Na^+$ solutions.

(i) If you mix equal volumes of the above two solutions to prepare 1 L of solution, calculate the pH of the resultant buffer solution. K_a for acetic acid = 1.8×10^{-5}

(ii) If you add 0.04 mol of NaOH to the above buffer solution, calculate the new pH of the buffer.

(iii) Giving necessary balanced chemical equations explain the buffering action of the above buffer solution when a small amount of base is added.

(40 marks)

(c) Usually HCl solution is standardized against borax ($Na_2B_4O_7$). During a titration between HCl and borax, 0.1 M H_3BO_3 is formed at the equivalence point. If the Ka value of H_3BO_3 is 5.8×10^{-10} select suitable indicator(s) from the given list. Justify your answer by using a suitable calculation.

$$H_3BO_3 + H_2O \rightleftharpoons H_2BO_3^- + H_3O^+$$

Indictor	pKa
Thymol Blue	1.65
Bromocresol purple	6.28
Bromocresol green	4.66

(30 marks)

(d) Sketch the titration curve for the above part (c) titration if the HCl solution is in the titration flask. Clearly label the axes of your graph.

(10 marks)

(04) Answer **all** parts.(***show all the calculations with relevant to your answer for part (c) and (d)***)

(a) Define the following.

(i) Accuracy
(ii) Precision
(iii) Relative error
(iv) Outliers

(20 marks)

(b)

(i) Write two types of systematic errors that can be arisen during an experiment.
(ii) Briefly explain how you would correct the errors that you mentioned above in b (i).

(20 marks)

(c) Bioaccumulation of Cr content in a fresh water edible fish verity was determined and the results are shown in the following table.

Experiment Number	Cr Content (µg/g)
1	0.117
2	0.103
3	0.085
4	0.215
5	0.096

Identify any rejectable data point at the 95% confidence level.

(25 marks)

(d) Selenium content in experimental fish feed was determined using a new analytical method and resulting data are given in the following table.

Sample	Se content (m/g)
1	0.0432
2	0.0347
3	0.0430
4	0.0453
5	0.0472

(i) Calculate the coefficient of variation of the above data

(ii) What do you report the Se content as the 95% confidence level?

(35 marks)

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Note :

(i) Confidence limits are defined as $\mu = \bar{x} \pm \frac{ts}{\sqrt{N}}$ where μ is the sample mean and s is the standard deviation.

(ii) Qcrit = ($|Xq-Xn|/X_{highest}-X_{lowest}$) where Xq is the questionable result and Xn is the nearest neighbor of the questionable result.

(iii) Values of t for various levels of probability and Rejection quotient at different confidence limits are attached.

Values of *t* for Various Levels of Probability

Degrees of Freedom	80%	90%	95%	99%	99.9%
1	3.08	6.31	12.7	63.7	637
2	1.89	2.92	4.30	9.92	31.6
3	1.64	2.35	3.18	5.84	12.9
4	1.53	2.13	2.78	4.60	8.61
5	1.48	2.02	2.57	4.03	6.87
6	1.44	1.94	2.45	3.71	5.96
7	1.42	1.90	2.36	3.50	5.41
8	1.40	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
15	1.34	1.75	2.13	2.95	4.07
20	1.32	1.73	2.09	2.84	3.85
40	1.30	1.68	2.02	2.70	3.55
60	1.30	1.67	2.00	2.62	3.46
∞	1.28	1.64	1.96	2.58	3.29

Rejection Quotient, *Q*, at Different Confidence Limits[a]

No. of Observations	Confidence Level		
	Q_{90}	Q_{95}	Q_{99}
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
15	0.338	0.384	0.475
20	0.300	0.342	0.425
25	0.277	0.317	0.393
30	0.260	0.298	0.372