

RAJARATA UNIVERSITY OF SRI LANKA FACULTY OF APPLIED SCIENCES

B.Sc. (General) Degree in Applied Sciences Second year Semester II Examination – January/February 2023

CHE 2103 – ANALYTICAL CHEMISTRY

Time: - One (01) hour

Answer both questions.

The use of a non-programmable calculator is permitted.

- 1. a) Perform the following calculations to the correct number of significant figures:
 - (i). $27.5 \times 1.82 \div 100.04$

(ii).
$$\frac{(2.0944 + 0.0003233 + 12.22)}{7.001}$$

- b) Make the following calculations and find the uncertainties in the final result:
 - (i). $(12.384 \pm 0.002) + (7.82 \pm 0.04) (5.6 \pm 0.1)$

(ii).
$$\frac{(39.84 \pm 0.04)(0.0994 \pm 0.0001)(224.3 \pm 0.2)}{426.4 \pm 0.3}$$

- c) The concentration of a solution was determined by four separate titrations, the results obtained were 0.2041, 0.2049, 0.2039 and 0.2043. Calculate the mean, median, range, standard deviation, and coefficient of variation.
- d) A chemist determined the percentage of iron in an iron ore and the values are, \bar{x} =12.60, s = 0.10 n = 4. Calculate the mean values of the 90 % and 99 % confidence intervals. (Hint: student's t values for n= 4 at 90 % and 99 % confidence intervals are 2.353 and 5.841 respectively).
- e) A 0.1557 g sample is dissolved in water, any sulfate present in the sample is precipitated as BaSO₄ by adding Ba(NO₃)₂. After filtering and rinsing the precipitate, it is dissolved in 25.00 mL of 0.02011 M EDTA. The excess EDTA is then titrated with 0.01113 M, Mg²⁺, requiring 4.23 mL to reach the end point. Calculate the percentage of Na₂SO₄ (by w/w) in the sample.
- f) A sample of iron ore is weighing 0.6428 g is dissolved in acid, the iron is reduced to Fe²⁺, and the solution is titrated with 36.30 mL of 0.01753 mol L⁻¹ K₂Cr₂O₇ solution. Calculate the percentage of iron in the sample.

- g) Determine the percentages of Na and K in a 0.6128 g sample which contains NaCl and KCl, treated with AgNO₃ yielded 1.039 g of dried AgCl.
- h) A sample of estrogens was separated using o-dichlorobenzene as the mobile phase. Two components were found when the spots were formed. The solvent fronts and solute fronts are shown below for both standards and unknowns. Determine the possible estrogens based on the experiments listed below.

	Solvent front / mm	Solute front / mm		
Estradiol	78	8		
Equilenin	79	32		
Estrone	77	69		
Estriol	80	16		
Unknown 1	79	34		
Unknown 2	79	70		
		$(8 \times 15 = 120 \text{ marks})$		

2. Answer Part A or Part B

Part A

a) (i) The following replicate observations were obtained during a measurement and they are arranged in ascending order:

Check whether the observation 4.85 can reject as an outlier at 95% and 99% confidence levels?

(20 marks)

(Hint:-)

N	Qcrit	Qcrit	Qcrit	
	(CL:90%)	(CL: 95%)	(CL: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	

(ii) Use the method of least squares in obtaining the straight line for the calibration curve and predict the value of y if x = 78.

X	75	80	93	65	87	71	98	68	84	77
У	82	78	86	72	91	80	95	72	89	74

Hint:-m =
$$\frac{\sum x_{i}y_{i} - \sum x_{i} \sum y_{i}/n}{\sum x_{i}^{2} - (\sum x_{i})^{2}/n}$$

(40 marks)

b) A 0.200 g sample of pyrolusite is analysed for manganese content as follows. Add 50.0 mL of a 0.100 M solution of ferrous ammonium sulphate to reduce the MnO₂ to Mn²⁺. After reduction is complete the excess ferrous ion is titrated in acid solution with 0.0200 M KMnO₄, requiring 15.0 mL. Calculate the percent manganese in the sample as Mn₃O₄.

(20 marks)

Part B

- a) Define the following terms:
 - (i). Stationary phase.
 - (ii). Mobile phase.

(10 marks)

- b) Answer the following and construct a titration curve for the titration of 25.0 mL of 0.125 M NH_3 solutions with 0.0625 M HCl. (Ka and Kw values are 5.70×10^{-5} and 1.00×10^{-14} respectively).
 - (i). Calculate the volume of titrant needed to reach the equivalence point, and the pH value at the equivalence point.
 - (ii). Calculate the pH values before adding the titrant, addition of 10.0 mL, 30.0 mL, 60.0 mL and 80.0 mL of titrant.

(50 marks)

- c). A certain sample of phosphate rock contains 24.24% P_2O_5 . A 0.5216 g sample is analyzed by precipitating $Mg(NH_3)PO_4.6H_2O$ and igniting the precipitate to $Mg_2P_2O_7$. Calculate the weights to obtain of:
 - (i). MgNH₄PO₄.6H₂O.
 - (ii). Mg₂P₂O₇.

(20 marks)

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