-1.14



RAJARATA UNIVERSITY OF SRI LANKA FACULTY OF APPLIED SCIENCES

B.Sc. Second year Semester II Examination – November / December 2016 CHE 2103 –Analytical Chemistry I

Answer both questions.

Time: 1 hour

The use of non-programmable calculator is permitted.

- 1). (a) Carry out the following operations and express each answer with the correct number of significant figures:
 - (i) $2.64 \times 10^3 + 3.27 \times 10^2$
 - (ii) $\frac{0.0382 \times 3.65 \times 10^3 \times 2.304}{8.64 \times 10^4}$
 - (b) Calculate the mean and relative average deviation in ppt, for the following set of data: 0.0512, 0.0520, 0.0516, and 0.0506.
 - (c) What is meant by the term determinate error and briefly explain the different types of determinate errors and give one reason to occur in each type of determinate error.
 - (d) Use the Q test to determine if the 3.483 value can be dropped from the following data set at 90 % confidence level.

3.274, 3.258, 3.265, 3.258, 3.350, 3.483

Hint: - Values of Q at 90% confidence:

| Q | 0.94 | 0.74 | 0.64 | 0.56 | 0.51 | 0.47 | 0.44 | 0.41 |
|----------------|------|------|------|------|------|------|------|------|
| \overline{N} | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |

(e) Based on the data given (d) above, calculate the 95% confidence interval and report the statistically probable mean value at 95% confidence.

Hint:- Values of Student's t at 95% confidence

| t | 12.71 | 4.303 | 3.182 | 2.776 | 2.571 | 2.447 | i | | 2.262 | 2.228 |
|---------------------|-------|-------|-------|-------|-------|-------|---|---|-------|-------|
| Degrees of freedom. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |

- (f) Write both a charge balance and a mass balance equation for a 0.1M H₂SO₄. Assume the H₂SO₄ ionizes to both HSO₄ and SO₄-2.
- (g) A 3.2306 g iron ore sample is dissolved in acid. The iron is reduced to Fe²⁺ and the solution is diluted to 250 cm³ in a volumetric flask. 50 cm³ aliquots are titrated with 0.01753 mol dm⁻³ standard solution of K₂Cr₂O₇. The average titrant volume was 36.30 cm³ to reach the equivalence point.
 - (i) Write down the redox reaction for the titration.
 - (ii) Calculate the weight percent of iron (55.847) in the ore sample.
- (h) Express the weight percent in terms of Fe₂O₃ (FW 159.69) for the data given in part (f) above.
- (i) Lanthanum iodate, La(IO_3)₃, (F.W. 663.62) has K_{sp} =1.0×10⁻¹¹. How many grams lanthanum iodate will dissolve in 1.00 L pure water?
- (j) Estimate the amount (in gram) of lanthanum iodate will dissolve in 1.00 L of a 0.050 mol dm⁻³ solution of soluble LiIO₃? State any assumption made in this calculations.

 $(10 \times 12 \text{ marks} = 120 \text{ marks})$

2). Answer either part (A) or part (B)

Part (A)

Answer all parts.

(a) Gravimetric analysis of nickel by precipitation with dimethylglyoxime gave the linear (y = mx+b) calibration. Regression values for the slope and intercept, with their standard deviations are 0.297 ± 0.020 , 0.20 ± 0.035 respectively. Calculate the weight percentage of nickel, and estimate the error using propagation formulas, for a sample with precipitate weight 5.120 ± 0.001 g.

(25 marks)

(b) Calculate the variances of the data 3.274, 3.258, 3.265, 3.258, 3.350, 3.483 with and without the 3.483 value. Apply the F-test to determine if there is a significant difference in the two variances at 90% confidence. Does your F- test result agree with the Q test result?

Hint:- Values of F at 90% confidence

| Degrees | Degrees of freedom s ₁ | | | | | | | |
|---------------------------------|-----------------------------------|------|------|------|------|--|--|--|
| of freedom s ₂ | 2 | 3 | 4 | 5 | 6 | | | |
| 2 | 9.00 | 9.16 | 9.24 | 9.29 | 9.33 | | | |
| 3 | 5.46 | 5.39 | 5.34 | 5.31 | 5.28 | | | |
| 4 | 4.32 | 4.19 | 4.11 | 4.05 | 4:01 | | | |
| 5 | 3.78 | 3.62 | 3.52 | 3.45 | 3.90 | | | |
| 6 | 3.46 | 3.29 | 3.18 | 3.11 | 3.05 | | | |

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(30 marks)

(c) A 2.00 g sample of limestone was dissolved in hydrochloric acid and all the calcium present in the sample was converted to Ca²⁺_(aq). Excess ammonium oxalate solution, (NH₄)₂C₂O_{4(aq)}, was added to the solution to precipitate the calcium ions as calcium oxalate, CaC₂O_{4(s)}. The precipitate was filtered, dried and weighed to a constant mass of 2.43 g. Determine the percentage by mass of calcium in the limestone sample. (The relative atomic weight of C, O and Ca are 12.01, 16.00 and 40.08 respectively).

(25 marks)

Part (B)

Answer all parts.

(a) Draw the titration curve for 50 cm³ of 0.1 mol dm⁻³ Na₂CO₃ versus 0.1 mol dm⁻³ HCl. Explain why boiling the solution near the end point in this titration increases the sharpness of the end point.

(20 marks)

(b) Calculate the solubility of CaCO₃ solution using a systematic approach to chemical equilibrium. You may assume a closed system (no CO₂ (g) in atmosphere). (K_{a1} K_{a2}, K_w are 4.3×10⁻⁷, 4.8×10⁻¹¹ and 1.0×10⁻¹⁴ respectively).

(40 marks)

c) A student weighed out 2.950 g of FeSO₄.xH₂O and dissolved in 50 cm³ of sulphuric acid and poured into a 250 cm³ volumetric flask which was made up to the mark with distilled water. 25.0 cm³ of this solution was titrated and required 21.20 cm³ of 0.0100 mol dm⁻³ KMnO₄ solution to reach the end point. Estimate the x. (relative atomic weights of H , O, S and Fe are 1.08, 16.00, 32.07 and 55.85 respectively).

(20 marks)