70

(*University of Choice*)

MASINDE MULIRO UNIVERSITY OF SCIENCE AND TECHNOLOGY (MMUST)

MAIN CAMPUS

SPECIAL/SUPPLEMENTARY EXAMINATIONS

2021/2022 ACADEMIC YEAR

FIRST YEAR SECOND SEMESTER EXAMINATIONS

FOR THE DEGREE

OF

BSc (CHEMISTRY) AND BSc (INDUSTRIAL CHEMISTRY), BACHELOR OF EDUCATION (SCIENCE)

COURSE CODE: **SCH 120**

COURSE TITLE: **ANALYTICAL CHEMISTRY 1**

DATE: *Wednesday, 03/08/2022* **TIME:** *8.00 – 10.00 AM*

INSTRUCTIONS TO CANDIDATES

Answer all the Questions

TIME: 2 Hours

This Paper Consists of 5 Printed Pages. Please Turn Over.

QUESTION ONE [17 Marks]

a) Analytical chemistry employs a wide range of procedures, techniques, operations and equipment during laboratory work. Identify the procedure, technique, instrument or operation described below. [5 Marks]

(i) Sample preparation method in which the organic constituents are oxidized using various reagents such as nitric acid and sulfuric acid or a combination of reagents

(ii) Separation technique in which a liquid is separated from solid by carefully pouring off the liquid after allowing the mixture to settle.

(iii) The lowest analyte amount or concentration that can be detected by a given procedure and with a given degree of confidence

(iv) A simulated matrix that replaces a sample during a measurement, but maintaining the conditions identical to those under which a sample would be analyzed

(v) A sample is treated with a reagent to protect a functional group during a synthetic process

b) Some of the equipment in the modern Analytical Chemistry Laboratory has been designed very specialized use. State and briefly explain the specific uses of the following laboratory equipment. [3 Marks]

(i) Micropipette

(ii) Vacuum filtration equipment

(iii) Desiccator

c) You are provided with an analytical grade concentrated Nitric Acid (63.0 g /mol) which has a specific gravity of 1.42 and is 70.5% (w/w) HCl.

(i) Define the term "specific gravity" [1 Mark]

(ii) Calculate the concentration of the acid in moles per litre. [2 Marks]

(iii) Explain how you would make 250 mL of 0.50 M HNO_3 solution from the concentrated acid. (Show you calculations). [3 Marks]

d) Describe the preparation of the following. [4 Marks]

(i) 500 mL of 0.0750 M $AgNO_3$ from the solid reagent. (RFM = 169.872)

(ii) 1.00 L of 0.285 M HCl, starting with a 6.00 M solution of the reagent.

QUESTION TWO [13 Marks]

a) The reliability of the results from any Chemical Analysis depends on the quality control procedures put in place. One important procedure aimed at enhancing the reliability of analytical results is calibration of the analytical instruments and the use of standards.

(i) What is standard material? [1 mark]

(ii) State four qualities of a primary standard. [4 marks]

(iii) Define the term calibration as used in Analytical Chemistry. [1 Mark]

(iv) State the difference between an external standard and an internal standard. [2 Marks]

(v) Explain one advantage of using an internal standard over use of external standard [2 Marks]

b) Apart from use of standards, state three other ways in which errors can be minimized in analytical procedures. [3 Marks]

QUESTION 3 [21 Marks]

a) Standard solutions play a central role in all titrimetric methods

(i) What is a standard solution [I Mark]

(ii) State four characteristics of an ideal standard solution for a titrimetric method. [4 Marks]

(iii) A standard 0.0500M solution of K^+ is required to calibrate a flame photometric method to determine the element. Describe how 100 mL of this solution can be prepared from a primary standard K_2SO_4 (174.259 g/ mol) [3 Marks]

b) Analytical Chemistry students were required to determine the percentage of calcium in egg shells. They digested 10.0 g of crushed egg shells using 100.00 mL of 2.5 M $HNO_{3\,(aq)}$ which was in excess. The residues were filter off the solution and labeled W. From solution W, 25.00 mL were drawn, topped up to 100 mL using distilled water and labeled Q. During titration, 20.00 mL of Q required 23.50 mL of 0.2 M NaOH solution for complete neutralization.

(i) Write an equation for the reaction between nitric acid and egg shells. [1 Mark]

(ii) State one observation which will indicate that all the calcium in the eggshell had reacted with nitric acid. [1 Mark]

(iii) How will the students know that all the acid in the 20.0 mL of solution Q is completely neutralized by $NaOH_{(aq)}$ [1 Mark]

(iv) Calculate the concentration of nitric acid in solution Q. [2 Marks]

(v) Determine of moles of nitric acid in 100 mL of solution W. [3 Marks]

(vi) Calculate the percentage of calcium in the eggshell samples analyzed. [3 Marks]

(vii) Suggest two possible sources of error in the results obtained. [2 Marks]

QUESTION FOUR [19 Marks]

a) The results obtained for the determination of the ppm Ca in spring water using colorimetry method were: 3.92. 3.28, 4.18, 3.53 and 3.35. Determine whether the 4.18 ppm result is an outlier or should be retained at the 95% confidence level. [3 Marks]

b) A 0.1 M solution of acid was used to titrate 10 ml of 0.1 M solution of alkali and the following volumes (in mL) of acid were recorded:

9.38 9.68 9.73 9.89 9.71

(i) Calculate the 95% confidence limits of the mean.

(ii) Use the limits to decide whether there is any evidence of systematic error. [6 Marks]

c) A sample a mixture, containing only KCl and $BaCl_2$, weighing 0.235 g was dissolved in enough water and precipitated using excess $AgNO_3$ solution. This yielded 0.4637 g AgCl, dry weight. You required determine the percentage composition.

(i) Generate an expression for the amount of AgCl precipitated from NaCl [3 Marks]

(ii) Form an expression for the mass of AgCl precipitated from $Bacl_2$ [3Marks]

(iii) Hence determine the percentage of NaCl and $BaCl_2$ in the sample [4 Marks]

Good Luck

Critical Values of F at the 5% Probability Level (95% confidence level)

Degrees of Freedom (Denominator)	Degrees of Freedom (Numerator)								
	2	3	4	5	6	10	12	20	∞
2	19.00	19.16	19.25	19.30	19.33	19.40	19.41	19.45	19.50
3	9.55	9.28	9.12	9.01	8.94	8.79	8.74	8.66	8.53
4	6.94	6.59	6.39	6.26	6.16	5.96	5.91	5.80	5.63
5	5.79	5.41	5.19	5.05	4.95	4.74	4.68	4.56	4.36
6	5.14	4.76	4.53	4.39	4.28	4.06	4.00	3.87	3.67
10	4.10	3.71	3.48	3.33	3.22	2.98	2.91	2.77	2.54
12	3.89	3.49	3.26	3.11	3.00	2.75	2.69	2.54	2.30
20	3.49	3.10	2.87	2.71	2.60	2.35	2.28	2.12	1.84
∞	3.00	2.60	2.37	2.21	2.10	1.83	1.75	1.57	1.00

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

Critical Values for the Rejection Quotient, Q^*

Number of Observations	Q_{crit} (Reject if $Q > Q_{crit}$)		
	90% Confidence	95% Confidence	99% 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