MACHAKOS UNIVERSITY

University Examinations 2018/2019

SCHOOL OF PURE AND APPLIED SCIENCES

DEPARTMENT OF PHYSICAL SCIENCES

FIRST YEAR SECOND SEMESTER EXAMINATION FOR

BACHELOR OF SCIENCE IN ANALYTICAL CHEMISTRY

SAN 101: LABORATORY PROCEDURES AND CLASSICAL ANALYSIS

DATE: 7/5/2019 **TIME: 8.30-10.30 AM**

INSTRUCTIONS TO CANDIDATES

- The paper consists of **two** sections.
- Section **A** is **compulsory**.
- Answer any **two** questions from section **B**.

SECTION A: COMPULSORY

QUESTION ONE (30 MARKS)

a) Define the following terms as used in analytical chemistry.

i. Good laboratory practice (1 mark)

ii. Mother liquor (1 mark)

b) Explain the primary laboratory safety rule and implied responsibility to make it work. (2 marks)

c) State three essential attributes of a lab notebook. (3 marks)

d) List four properties of a primary standard. (4 marks)

e) Briefly explain one advantage and one disadvantage of using classical methods of analysis. (2 marks)

f) A linear calibration curve of corrected absorbance's plotted versus amount of protein gave a line of equation $y = 0.0163x + 0.0047$. Find the quantity of unknown protein that gives a measured absorbance of 0.264 when a blank has an absorbance of 0.095(3 marks)

g) Briefly explain four ways that you could apply to detect systematic error in an experiment. (6 marks)

h) The volume delivered by a buret is the difference between final and initial readings. If the uncertainty in each reading is ±0.03 calculate the uncertainty in the volume delivered. (3 marks)

i) 0.470 g of sample containing sodium carbonate was treated with 50 mL of 0.1M HCl. The excess HCl was back titrated using 0.1M standardized NaOH where 20 mls of NaOH was used. Calculate the percentage of sodium carbonate in the sample. (The molecular weight of sodium carbonate is 105.99 g). (5 marks)

SECTION B: ANSWER ANY TWO (2) QUESTIONS (20 marks each)

QUESTION TWO (20 MARKS)

a) Differentiate between the following terms

i. Molarity and molality (2 marks)

ii. Complexometric analysis and volumetric analysis (2 marks)

b) A typical seawater contains 2.7 g of salt (sodium chloride, NaCl) per 100 mL. Calculate.

(i) The molarity of NaCl in the sea water. (2 marks)

(ii) The grams of $MgCl_2$ present in 25 mL of seawater given that $MgCl_2$ has a concentration of 0.054 M in the ocean. (2 marks)

c) A sample of calcium carbonate is dissolved with 20.00 mL of 0.2254 M hydrochloric acid and the excess acid is titrated with 0.1041 M sodium hydroxide. After dissolution, a mass of 0.2719 g of the calcium carbonate sample requires a titer of 12.39 mL of sodium hydroxide to reach a phenolphthalein end point. Calculate the %w/w of $CaCO_3$ in the sample. (4 marks)

d) 32.50 g of the impure magnesium sulfate is dissolved in water and the solution is made up to 500.0 mL in a volumetric flask. Different volumes of 0.100 M $BaCl_2$(aq) are added to six separate 20.00 mL samples of this solution. This precipitates the sulfate ions as barium sulfate. The precipitate from each sample is filtered, rinsed with de-ionised water and then dried to constant mass. The results of this analysis are shown:

Results

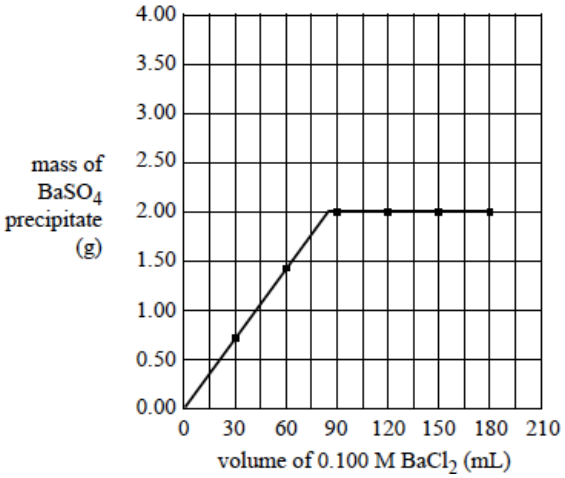
Mass of impure magnesium sulfate = 32.50 g

Volume of volumetric flask = 500.0 mL

Volume of magnesium sulfate solution in each sample = 20.00 mL

Sample	1	2	3	4	5	6
volume of $BaCl_2$(aq) added (mL)	30.0	60.0	90.0	120	150	180
mass of $BaSO_4$(s) precipitated (g)	0.704	1.41	2.00	2.00	2.00	2.00

These results are shown on the graph below.



i. Indicate the analytical term used to describe this classical method of analysis. (1 mark)

ii. Write a balance chemical equation for the reaction that precipitates sulfate ions as barium sulfate. (1 mark)

iii. Explain the importance of rinsing the precipitate with de-ionised water before drying. (1 mark)

(i) Explain why the amount of $BaSO_4(s)$ precipitated remains constant for the last four samples tested even though more $BaCl_2(aq)$ is being added. (2 marks)

(ii) Calculate the amount, in mole, of $SO_4^{2-}(aq)$ in the 500.0 mL volumetric flask. (3 marks)

(iii) Calculate the percentage, by mass, of magnesium sulfate in the powder. (2 marks)

QUESTION THREE (20 MARKS)

a) Differentiate between the following terms as used in Analytical Chemistry.

i. Precision and accuracy (2 marks)

ii. Random and Systematic error (2 marks)

b) Explain in details how you would carry out an analysis to determine if garden soil within Machakos University is contaminated with heavy metals. (7 marks)

c) In gravimetric analysis, one needs enough product to weigh accurately. Each tablet provides approximately 15 mg of iron. How many tablets should one analyze to provide 0.25 g of Fe_2O_3 products (2 marks)

d) A solution with a final volume of 500 mL was prepared by dissolving 25 mL of methanol (CH_3OH, density = 0.7914 g/mL in chloroform)

i. Calculate the molarity of methanol in the solution. (2 marks)

ii. The solution has a density of 1.454 g/mL. Calculate the molality of methanol. (2 marks)

e) A student prepared a 0.250 M NH_3 solution by diluting 8.45 (±0.04) of 28.0 (±0.5) wt% NH_3 [density = 0.899 (±0.003) g/mL] up to 500.0 (±0.2) mL. Determine the uncertainty in0.250 M. The molecular mass of NH_3, 17.030 5 g/mol, has negligible uncertainty relative to other uncertainties in this problem. (3 marks)

QUESTION FOUR (20 MARKS)

a) Differentiate between the following terms as used in analytical Chemistry

(i) Bouyancy and parallax. (2 marks)

(ii) Method blank and reagent blank. (2 marks)

b) From previous measurements of a low concentration of analyte, the signal detection limit was estimated to be in the low nanoampere range. Signals from seven replicate samples with a concentration about three times the detection limit were 5.0, 5.0, 5.2, 4.2, 4.6, 6.0, and 4.9 nA. Reagent blanks gave values of 1.4, 2.2, 1.7, 0.9, 0.4, 1.5, and 0.7 nA. The slope of the calibration curve for higher concentrations is m = 0.0229 nA/μM. Given that the standard deviation (s) is 0.56 nA. Calculate;

i. The signal detection limit. (2 marks)

ii. The minimum detectable concentration. (2 marks)

c) A pure compound called "tris" is used as a primary standard to measure concentrations of acids. The volume of acid required to react with a known mass of tris tells us the concentration of the acid. Calculate the true mass of tris if the apparent mass weighed in air is 100.00 g. (3 marks)

d) Briefly explain the following with regards to good laboratory practices

i. Equipment use. (3 marks)

ii. Receipt handling sampling and storage. (3 marks)

iii. Storage and retention of records and materials. (3 marks)

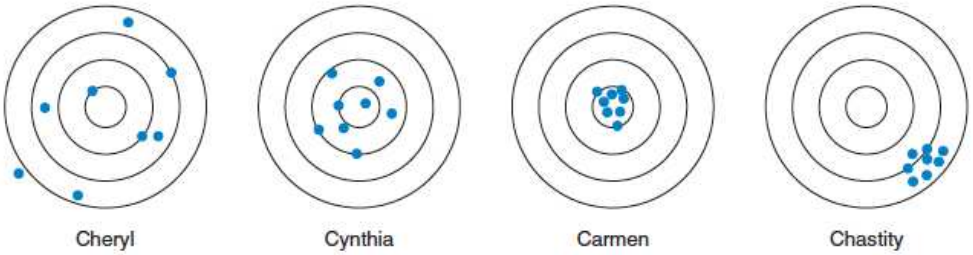
QUESTION FIVE (20 MARKS)

a) Using a relevant example, define the following terms

i. Internal standard (1 mark)

ii. Detection limit (1 mark)

b) Cheryl, Cynthia, Carmen and Chastity shot the targets at Girl Scout camp. Match each target shown below with the proper description.



i. Accurate and precise. (1 mark)

ii. Accurate but not precise. (1 mark)

iii. Precise but not accurate. (1 mark)

iv. Neither precise nor accurate. (1 mark)

c) A solution of ammonia in water is called "ammonium hydroxide" because of the equilibrium:

$NH_3 + H_2O \rightleftharpoons NH_4^+ + OH^-$

Given that the density of concentrated ammonium hydroxide, which contains 28.0 wt% NH_3 is 0.899 g/mL. Determine volume of this reagent should be diluted to 500.0 mL to make 0.250M. (4 marks)

d) Serum containing Na^+ gave a signal of 4.27 mV in an atomic emission analysis. Then 5.00 mL of 2.08 M NaCl were added to 95.0 mL of serum. This spiked serum gave a signal of 7.98 mV. Find the original concentration of Na^+ in the serum. (5 marks)

e) In a preliminary experiment, a solution containing 0.083 7 M *X* and 0.066 6 M *S* gave peak areas of $A_X = 423$ and $A_S = 347$. To analyze the unknown, 10.0 mL of unknown, and the mixture was diluted to 25.0 mL in a volumetric flask. This mixture gave the chromatogram shown, for which $A_X = 553$ and $A_S = 582$. Find the concentration of *X* in the unknown. (5 marks)