TECHNICAL UNIVERSITY OF MOMBASA

FACULTY OF APPLIED AND HEALTH SCIENCES

DEPARTMENT OF MEDICAL SCIENCES

UNIVERSITY EXAMINATION FOR:

DIPLOMA IN PHARMACEUTICAL TECHNOLOGY

APM 2308: ANALYTICAL PHARMACEUTICAL CHEMISTRY

END OF SEMESTER EXAMINATION

SERIES: AUGUST 2019

TIME: 4HOURS

DATE: Pick DateSelect MonthPick Year

Instructions to Candidates

You should have the following for this examination

examination pass and student ID

This paper consists of threeSection(s). Attempt ALL QUESTIONS in section A and B and ANY one question in section C .**use the answer booklets**

SECTION A (40 MARKS)

Question ONE

The following have been supplied to you

1.0M NaOH

0.1M HCl

6 (six) Acetylsalicylic acid (ASA) tablets

Mortar and Pestle

2 pipettes (25.0 ml)

Burette

Volumetric flask

3 Conical

Phenolphthalein indicator

Distilled Water

Any other apparatus and reagents required for the experiment

AIM: Assay of Acetylsalicylic acid in 300mg ASA tablets B.P

Procedure:

1. Weigh six ASA tablets

Mass of six ASA tablets:

2. Triturate ALL the tablets in a mortar using the pestle and weigh as accurately as possible 0.5g of powder

Mass of powder weighed

3. Transfer the powder quantitatively into a conical flask and add 30 ml of distilled water

4. Pipette 25.0 ml of 1M NaOH solution (**excess**) into the mixture and boil for 10 minutes

5. Cool the mixture (can use cold tap water)

6. Transfer the mixture quantitatively into a 250 ml volumetric flask, add distilled water to the mark

Mix the contents well and label the solution

7. Pipette 25.0 ml of the labeled solution and titrate with 0.1 M HCl solution

Repeat the titration twice

(a) Tabulate the results (10 marks)

(b) Write a balanced equation on the reaction of acetylsalicylic acid and NaOH (2 marks)

(c) Write a balanced equation on the reaction of HCl and NaOH (1mark)

(d) Calculate:

(i) Moles of HCl that reacted with NaOH (2 marks)

(ii) Moles of excess NaOH in 25 ml reaction mixture (2 marks)

(iii) Moles of excess NaOH in 250 ml reaction mixture (2 marks)

(iv) Moles of NaOH initially present before hydrolysis (2 marks)

(v) Moles of NaOH that actually reacted with acetyl salicylic acid (2 marks)

(vi) Moles of acetylsalicylic acid in 250 ml solution (2 marks)

(vii) Weight of acetylsalicylic acid in the weighed powder (2 marks)

(viii) Acetylsalicylic acid content per tablet (in milligrams) (4 marks)

(ix) The B.P specification is 99.5-101.0% acetylsalicylic acid content of the label claim.

Do the ASA tabs pass? (5marks) (show **your working**)

SECTION B (40 MARKS)

Question TWO (20 marks)

a) Define the following terms as used in pharmaceutical analysis

i. Primary standard
ii. Secondary standard
iii. Non-aqueous titration
iv. a buffer
v. Partition coefficient (10 Marks)

(b) Briefly discuss (10 Marks)

i. Features of a primary standard
ii. Theory of colored indicators as used in acid-base titrations
iii. Qualitative/ quantitative analysis
iv. Steps followed in a typical quantitative analysis
v. Evaluation of analytical data

Question THREE

(a) Explain how you would prepare (5 Five) litres of sodium carbonate solution from the stock powder (5 marks)

Na=23 C=12 O=16

(b) A sample containing 25.14g of neutral salts, glucose and sodium carbonate/bicarbonate buffer was dissolved in 100 ml of water. A 25 ml aliquot of the resultant solution required 20.35 ml of 0.0987M HCl when titrated to the phenolphthalein end point. A second 25 ml aliquot was titrated to the methyl orange end point and required 56.75 ml of the acid.

i. Sketch a titration curve for the titration (3 Marks)
ii. Calculate the percentage of Na_2CO_3 and $NaHCO_3$ in the sample (12 Marks)

SECTION C (20marks)

ATTEMPT ANY ONE QUESTION

Question FOUR

(a) 25.0 cm^3 of a solution containing a mixture of sodium carbonate and sodium hydrogen carbonate were titrated with 0.11M aqueous hydrochloric acid using phenolphlaein and methyl orange indicators. The following results were obtained

		Volume of 0.11M HCl used			
Volume of mixture used (cm^3) portions (Na_2CO_3 and $NaHCO_3$	Indicator	Trial	1st	2nd	3rd
25.0	Phenolpthalein	11.50	11.00	11.05	10.95
25.0	Methyl orange	39.0	38.85	38.80	37.75

(i) Calculate the average volume used in the phenolphthalein titration (1 mark)

(ii) Write an equation for the reaction that takes place between the mixture and HCl in the phenolphthalein titration and explain your answer (4 marks)

(iii) Calculate the average volume used in the methyl orange titration (1 mark)

(iv) Write an equation for the reaction that takes place between the mixture and HCl in the methyl orange titration and explain your answer (3 marks)

(v) Determine the volume of HCl which reacted with sodium carbonate and sodium hydrogen carbonate (4 marks)

(vi) Calculate the moles per liter and grams per liter of sodium carbonate and sodium hydrogen carbonate in the mixture (5 marks)

(vii) Calculate the % proportion of each component in the mixture (2 marks)

Na= 23 H= 1 C= 12 O= 16

Question FIVE

(a) Calculate the p^H of the following solutions

(i) 0.1 M Formic acid ($Ka = 1.77 \times 10^{-4}$)

(ii) 0.005 M HCl

(iii) 0.1 M Ammonia ($K_b = 1.8 \times 10^{-5}$)

(5 marks)

(b) Determine the percentage of ionization of acetic acid at p^H (pKa acetic acid = 4.76)

(i) 3.76

(ii) 5.76

(5 marks)

(c) (i) Discuss preparation of 0.1 M Ammonium chloride buffer with pH =9.0 (2 marks)

(ii) Calculate the concentration of the following in the prepared buffer (pKa ammonia = 9.25)

I. NH_3

II. NH_4^+

(8 marks)

[Mwt N= 14, H = 1, Cl=35]