



**TECHNICAL UNIVERSITY OF MOMBASA**

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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<sub>2019</sub>

**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**

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## SECTION A (40 MARKS)

### Question ONE

*The following have been supplied to you*

*Solution A contains a mixture of sodium chloride and sodium hydroxide*

*Solution B contains 0.1M standard hydrochloric acid solution*

*Solution C contains 1.6g/L silver nitrate*

*1M nitric acid*

*Potassium chromate (IV) indicator*

*Calcium carbonate powder*

*2 burettes*

**Na =23, Cl =35, H =1, O =16, Ag =108, N =14, I =127, C =12**

**AIM: To determine the percentage by mass of sodium chloride and sodium hydroxide in the mixture**

**Procedure:**

#### Part 1

- (a)
- Pipette 25.0 ml of aliquots of solution A into a conical flask and add three drops methyl orange indicator
  - Fill the burette with solution B
  - Carry out the titration to the end point
  - Repeat the titration three more time
  - Tabulate the results **10 Marks**
- (b) Calculate the average volume of 0.1M HCl used **1 Mark**
- (c) Write an equation on the reaction of HCl and the substance in solution A **2 marks**
- (d) Calculate the mass concentration (grams per liter) of the substance that reacted in solution A **6 Marks**
- (e) Name the type of titration above **1 Mark**

#### Part II

- (a)
- Pipette 25.0 ml of solution A into a clean conical flask
  - Add 5 ml of 1.0M nitric acid followed by the addition of powdered calcium carbonate, little at a time and with swirling until some solid remains undissolved
  - Add three drops of potassium chromate (VI) indicator to the solution in the conical flask
  - Fill second burette with solution C
  - Carry out the titration to the first permanent red-brown end point
  - Repeat the titration three times and tabulate your observations **10 Marks**

- (b) Calculate the average volume of solution C used in the titration **1 Mark**
- (c) Write an equation for the reaction between solution C and the substance that reacted in solution A **2 Marks**
- (d) Calculate the mass concentration (grams per liter) of the substance that reacted in solution A **3 Marks**
- (e) From results of part I and part II above, calculate the percentage by mass of each compound in the mixture (solution A) **3 Marks**
- (f) Name the type of titration in part II **1 Mark**

## **SECTION B (40 MARKS)**

### **Question TWO** (20 marks)

a) A quality control pharmacist took a sample of iodine solution manufactured by Tum pharmaceuticals and analysed it by titration with standard 0.01M sodium thiosulfate solution. 10.0 ml of the sample was pipetted and diluted with distilled water to 250.0 ml in a volumetric flask. 25.0 of the aliquot of the diluted solution reacted completely with 19.69 ml of 0.01M sodium thiosulfate in presence of starch indicator

- Write a balanced half and full ionic equation for the reaction between iodine and thiosulfate
- Calculate the number of moles of thiosulfate ions present in the titrant
- Calculate the number of moles of iodine in 25.0 ml of the dilute solution
- Calculate number of moles of iodine in 250.0 ml of dilute solution
- What is the number of moles of iodine in 10.0 ml of the sample
- Calculate the mass of iodine in grams in 10.0 ml sample
- Calculate the percentage weight-volume of iodine in the sample

### **Question THREE** (20 marks)

A member of DPT 16S was on attachment in a pharmaceutical factory QA department and was assigned to assay acetylsalicylic acid in 300mg tablets B.P. She proceeded as follows

- Five tablets were weighed and the weight noted to be 1.8g
- All the tablets were crushed in a mortar and 0.5g of the powder transferred into a conical flask with 30 ml of distilled water
- 25.0 ml of 1.0M sodium hydroxide (excess) were added and the mixture boiled for 15 minutes to hydrolyze the aspirin completely
- The hydrolyzed mixture was cooled and transferred into a 250 ml volumetric flask and diluted with distilled water to the mark. The contents were mixed well.
- 25.0 ml portion of the diluted reaction mixture was titrated with 0.005M sulphuric acid with phenolphthalein as the indicator. The average volume of sulphuric acid used was 20.40 ml
  - Write a balanced equation for the reaction of acetylsalicylic acid with sodium hydroxide

- b. Write a balanced equation of the reaction of excess sodium hydroxide and sulphuric acid
- c. Calculate
- Moles of sulphuric acid that reacted with sodium hydroxide **2 Marks**
  - Moles of excess sodium hydroxide in 25.0 ml portion **2 Marks**
  - Moles of excess sodium hydroxide in 250 ml portion **2 Marks**
  - Moles of sodium hydroxide initially present before hydrolysis **2 Marks**
  - Moles of sodium hydroxide that actually reacted with acetylsalicylic acid **2 Marks**
  - Moles of acetylsalicylic acid in 250 ml solution **2 Marks**
  - Weight of aspirin in 250 ml solution (0.5g) of powder **3 Marks**
  - Acetylsalicylic acid content per tablet (in milligrams) **3 Marks**
  - The percentage of the acetylsalicylic acid content compared to the label claim of 300 mg per tablet **2 Marks**

**SECTION C (20marks)**

ATTEMPT ANY ONE QUESTION

**Question FOUR**

10.0grams of a sample of limestone were dissolved in 200 ml of 1M hydrochloric acid. The resulting solution was made to 1000 ml with distilled water. 25 ml of this solution required 41.0 ml of 0.005M sodium hydroxide solution for complete neutralization. Assume all basic material in limestone is calcium carbonate

- (a) Write a balanced equation for the following reactions **3 Marks**
- Calcium carbonate and hydrochloric acid
  - Hydrochloric acid and sodium hydroxide
- (b) Calculate the moles of excess hydrochloric acid in 25.0 ml solution titrated with sodium hydroxide **3 Marks**
- (c) Calculate moles of excess hydrochloric acid in 1000 ml prepared **3 Marks**
- (d) Determine the actual moles of hydrochloric acid that reacted with calcium carbonate **4 Marks**
- (e) Determine the moles of calcium carbonate in 1000 ml and hence the mass of calcium carbonate **4 Marks**
- (f) Calculate the percentage of calcium carbonate in the sample **3 Marks**

C = 12 O = 16 Ca = 40

**Question FIVE**

0.3g of impure zinc dust was reacted with excess of iron (III) sulphate in the presence of dilute sulphuric acid. The iron (II) sulphate produced required 17.10 ml of 0.1M potassium permanganate (VII) ( $\text{KMnO}_4$ ) for complete reaction

$Zn = 65.4$

- a. Write a balanced half ionic and full ionic equation for the reaction between zinc dust ( $Zn_s$ ) and iron (III) ions  $Fe^{3+}_{aq}$  **2 Marks**
- b. Write a balanced full equation for the reaction between iron (II) ions ( $Fe^{2+}$ ) and manganite ions ( $MnO_4^-$ ) in presence of sulphuric acid **2 Marks**
- c. Determine the stoichiometric ratio of  $Zn_s$  and  $MnO_4^-$  ions **2 Marks**
- d. Calculate **12 Marks**
  - i. Moles of  $MnO_4^-$  that reacted
  - ii. Moles of  $Zn_s$  that reacted with  $MnO_4^-$
  - iii. Mass of  $Zn_s$  in the dust
  - iv. The percentage purity of the zinc
- (e) Explain why excess iron (III) sulphate is used in this reaction **2 Marks**