



**UNIVERSITY OF RUHUNA – FACULTY OF ALLIED HEALTH SCIENCES**

**DEPARTMENT OF PHARMACY**

**THIRD BPHARM PART I EXAMINATION – JANUARY 2023**

**PH 3113 ANALYTICAL CHEMISTRY - SEQ PAPER**

**TIME: TWO HOURS**

**INSTRUCTIONS**

- There are **four** questions in part A, B and C in this SEQ paper.
- Answer each part in separate booklets provided.
- No paper should be removed from the examination hall.
- Do not use any correction fluid.
- Use illustrations where necessary.

**PART A**

- 01.
- 1.1 Define the term “pharmaceutical impurities” by giving two relevant examples. *(15 marks)*
- 1.2 List down the principles for fixing the limits for impurities. *(20 marks)*
- 1.3 Briefly explain what is meant by the “permissible limits of impurities”. *(20 marks)*
- 1.4 Sometimes it is required to revalidate analytical procedures. Name such instances. *(15 marks)*
- 1.5 “Reference standards are an important analytical tool in industry and quality control laboratories.” Justify this statement. *(30 marks)*

**PART B**

- 02.
- 2.1 A 0.5664 g ore sample containing aluminum was dissolved in nitric acid and then filtered. The solution was made basic with ammonium hydroxide to precipitate  $\text{Al}(\text{OH})_3$  (FW 78.004). This was filtered in a porous glass crucible, rinsed with dilute ammonium hydroxide, ignited, cooled in a desiccator, and weighed. The resulting alumina,  $\text{Al}_2\text{O}_3$  (FW 101.94), weighed 0.1605 g.
- 2.1.1 Why is the solution filtered after acid dissolution? *(05 marks)*
- 2.1.2 Why rinse with ammonium hydroxide solution? *(05 marks)*
- 2.1.3 What chemical transformation takes place during ignition? (Show the balanced chemical equation). *(10 marks)*
- 2.1.4 Why use a desiccator during cooling? *(05 marks)*
- 2.1.5 Calculate the weight percent Al (AW 26.9815) in the sample. *(25 marks)*

2.1.6 Suggest an alternative method that produces non-gelatinous, easily filtered, spherical particles (hint: homogenous precipitation method). **(10 marks)**

2.2 A 0.2500 g mixture of magnesium oxide (FW 40) and calcium oxide (FW 56) **only** was dissolved in dilute nitric acid and neutralize the mixture with dil. NaOH and the solution mixture was made up to 1.00 dm<sup>3</sup> with distilled water. A 25.00 cm<sup>3</sup> portion of this solution was buffered (pH 10) and after addition of the indicator, was titrated against 0.0100 mol dm<sup>-3</sup> EDTA solution, 12.90 cm<sup>3</sup> titrant was required.

2.2.1 Write chemical reactions between EDTA and Mg<sup>2+</sup> and Ca<sup>2+</sup> ions. **(10 marks)**

2.2.2 Find the percentage by mass of magnesium oxide and calcium oxide in the mixture. **(30 marks)**

03.

3.1 There are three different methods in argentometric titration.

3.1.1 Giving chemical equations, explain briefly the steps involved in Volhard's method of argentometric titration. **(20 marks)**

3.1.2 The monochloroacetic acid (ClCH<sub>2</sub>COOH, MW 94.5) preservative in a 100.00 cm<sup>3</sup> sample of carbonated beverage was extracted into diethyl ether and then returned to aqueous solution as ClCH<sub>2</sub>COO<sup>-</sup> with 1.0 mol dm<sup>-3</sup> NaOH. This aqueous extract was acidified and treated with 50.00 cm<sup>3</sup> of 0.0452 mol dm<sup>-3</sup> AgNO<sub>3</sub> where the following reaction occurred:



After filtration of the AgCl(s) titration of the filtrate and washing required 10.43 cm<sup>3</sup> of an NH<sub>4</sub>SCN solution. Titration of a blank (just distilled water) taken through the above entire process required 22.98 cm<sup>3</sup> of the same NH<sub>4</sub>SCN solution.

Calculate the weight in mg, of ClCH<sub>2</sub>COOH the in the beverage sample. **(30 marks)**

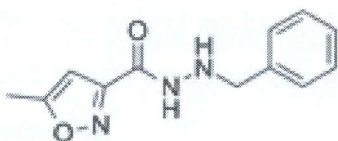


3.2 The most common method for the determination of pharmaceutical bases is the direct titration with perchloric acid in glacial acetic acid. In the preparation of a solution of 0.1 M perchloric acid, the added amount of acetic anhydride should be optimum. Explain why?

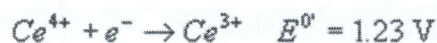
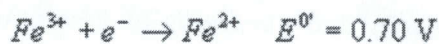
(10 marks)

3.3 Propose the most suitable titrimetric method for the assay of the following drug isocarboxazid and briefly explain the principle of the titrimetric method that you proposed.

(15 marks)



3.4 A 25.00 cm<sup>3</sup> portion of a Fe<sup>2+</sup> solution was titrated with 0.0200 Ce<sup>4+</sup> titrant. Both were in 1M HCl. The formal potentials of the redox reactions involved are:



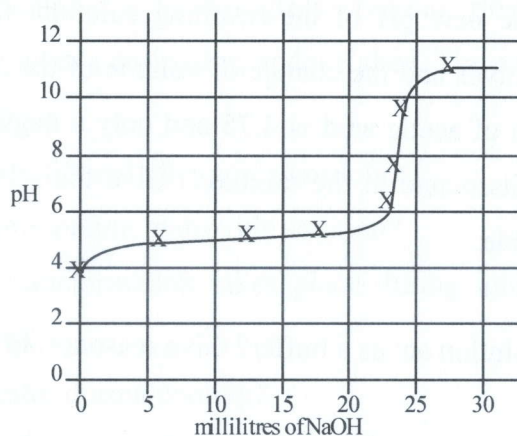
3.4.1 What is the potential half-way to the equivalence point? (10 marks)

3.4.2 What is the potential at the equivalence point? (10 marks)

3.4.3 What potential is produced at twice the volume of titrant required to reach the equivalence point? (05 marks)

04.

4.1 A 30.00 mL sample of a weak monoprotic acid was titrated with a standardized solution of NaOH. The pH measured after each increment of NaOH was added, and the following titration curve was obtained.



4.1.1 Explain how this curve could be used to determine the molarity of the acid and the dissociation constant  $K_a$  of the weak monoprotic acid. (20 marks)

4.1.2 If you were to repeat the titration using an indicator, which of the following indicator(s) should you select? Give the reason for your choice. (10 marks)

Methyl red ( $K_a = 1 \times 10^{-5}$ ), Cresol red ( $K_a = 1 \times 10^{-8}$ ), Alizarin yellow ( $K_a = 1 \times 10^{-11}$ )

4.1.3 Sketch the titration curve that would result if the weak monoprotic acid was replaced by a strong monoprotic acid, such as HCl of the same molarity. Identify differences between this titration curve and the curve shown above. (20 marks)

### PART C

4.2

4.2.1 Define the terms *molarity* and *normality* of a solution used in the expression of concentration of pharmaceutical solutions. (08 marks)

4.2.2 Calculate the molarity and normality of  $H_2SO_4$  acid in a mixture of  $250.00 \text{ cm}^3$  of  $0.0500 \text{ mol dm}^{-3}$   $H_2SO_4$  acid and  $250.00 \text{ cm}^3$  of  $0.0010 \text{ mol dm}^{-3}$  of NaOH diluted up to  $1.00 \text{ dm}^3$  with distilled water. Give the relevant chemical equation(s) wherever applicable. (12 marks)

4.2.3 Calculate the pH of the above solution. (04 marks)

4.2.4 If you dissolve 3.2812 g of  $CH_3COONa$  (MW=82.03 g/mol) in the above solution in 4.2.2 calculate the new pH of the resulting solution. (Assume that all the solid  $CH_3COONa$  dissolves and the change of volume of the solution is negligible. Also consider that pKa of acetic acid is 4.75 and only a negligible amount of produced acetic acid will dissociate in the mixture.) Give the relevant chemical equation(s) wherever applicable. (20 marks)

4.2.5 Will the above solution act as a buffer? Give reason(s) to justify your answer. (06 marks)

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