(4) Index No:.....

# UNIVERSITY OF RUHUNA – FACULTY OF ALLIED HEALTH SCIENCES

## DEPARTMENT OF PHARMACY

## THIRD BPHARM PART I EXAMINATION – APRIL /MAY 2021

## PH 3113 ANALYTICAL CHEMISTRY – SEQ

**TIME: TWO HOURS**

---

**INSTRUCTIONS**

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

## PART A

**01.**

1.1

1.1.1 Define the term "Pharmaceutical impurities". (***05 marks***)

1.1.2 Name **two** relevant examples for pharmaceutical impurities. (***10 marks***)

1.1.3 Briefly explain the different types of pharmaceutical impurities and their consequences of impurities in the pharmaceutical Industry. (***25 marks***)

1.1.4 Write a short note on "the limit test for heavy metals". (***25 marks***)

1.2

1.2.1 List the characteristics that you would consider for the validation of an assay test. (***15 marks***)

1.2.2 Explain the term 'precision' in relation to analytical method validation. (***20 marks***)

## PART B

**02.**

2.1 Ethylenediamminetetracetic acid (EDTA) and Eriochrome Black T (EBT) are widely used in complexometric determination of several metal ions.

2.1.1 What are the advantages of using EDTA as a complexing agent (titrant)? (***10 marks***)

2.1.2 Briefly explain the underline principal for the detection of the end point of an EDTA titration with a metal ion using EBT as the indicator. Give the relevant chemical equations wherever necessary.

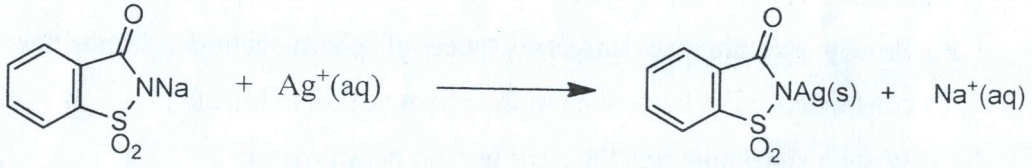
(You may use common abbreviations, $M^{2+}$, $Y^{4-}$ and $HIn^{2-}$ for a divalent metal ion, EDTA and EBT indicator respectively). (***20 marks***)

2.1.3 A student was given an impure zinc salt in order to determine the percentage of Zn. He dissolved 0.7100 g of the given salt in 25.00 mL of distilled water and the pH of the solution was adjusted to 10. It was titrated with 0.0160 *mol* $dm^{-3}$ EDTA solution using EBT as the indicator and obtained the average volume of 21.00 mL from triplicate readings for the end point. Determine the gram percentage of Zn in this sample. Assume that Zn is the only metal ion which reacts with EDTA in this sample (atomic mass of Zn is 65.38g). ***(30 marks)***

2.2 Argentometric titrations are based on the use of silver ion as a precipitating agent.

2.2.1 What are the two types of indicators that are employed in argentometric titrations? Give an example for each type with appropriate reaction(s). ***(20 marks)***

2.2.2 A 20-tablet sample of soluble saccharin was treated with 20.00 mL of 0.0818 *mol* $dm^{-3}$ $AgNO_3$. After removal of the solid, titration of the filtrate and washings required 2.81 mL of 0.0412 *mol* $dm^{-3}$ KSCN. Calculate the average number of milligrams of saccharin (205.17 g/mol) in each tablet. The reaction of saccharin with Ag+ ion is shown below: ***(20 marks)***




**03**. Give the reason(s) for the following:

3.1 In gravimetric analysis we have some control on particle size of the precipitate based on how we add reagents. ***(20 marks)***

3.2

3.2.1 Addition of acetic anhydride in the preparation of a standard acetous $HClO_4$ acid solution. ***(10 marks)***

3.2.2 Addition of mercuric acetate in the assay of **Amitriptyline Hydrochloride** in non-aqueous medium. ***(15 marks)***

3.2.3 Each ml of 0.1 M sodium methoxide is equivalent to 0.01412 g of Ethosuximide, $C_7H_{11}NO_2$ (FW= 141.17 g/mol) in titrimetric assay of it. ***(15 marks)***

3.3 Starch iodide paper or paste is used in the assay of sulphonamide drugs by diazotization titrations. ***(20 marks)***

3.4 The cathode potential at the equivalence point for the redox titration of $Fe^{2+}$ with $Ce^{4+}$is 1.26 V. In 1 M $HClO_4$, the formal potential for the reduction of $Fe^{3+}$ to $Fe^{2+}$ is + 0.77 V, and the formal potential for the reduction of $Ce^{4+}$ to $Ce^{3+}$is + 1.70 V. ***(20 marks)***

(5)

**04.**

4.1. Distinguish between the end point and equivalence point of a titration. (***10 marks***)

4.2 Consider the following two titrations with the base as the titrant:

**A-** HCl (0.1000 *mol* $dm^{-3}$) and NaOH (0.1000 *mol* $dm^{-3}$)

**B-** HAc (0.1000 *mol* $dm^{-3}$, pKa = 4.75) and NaOH (0.1000 *mol* $dm^{-3}$)

4.2.1 On the same graph, sketch titration curves for **A** and **B** titrations. (***15 marks***)

4.2.2 Describe all of the characteristics that can be seen within two curves. (***15 marks***)

4.2.3 Would the indicator bromocresol green, with a transition range of pH 3.8-5.4 be useful in both titrations? (***10 marks***)

## PART C

4.3 The pH of a solution can be maintained by using a buffer.

4.3.1 What are the **two** factors that determine the pH of a buffer? (***05 marks***)

4.3.2 Explain briefly how buffers maintain the pH on the addition of small amounts of $OH^-$ ions? (***10 marks***)

4.3.3 An extracellular fluid consists of bicarbonate buffer system, which made from carbonic acid and sodium bicarbonate. Discuss briefly how it maintains the pH on small addition of a strong acid and a strong base. (***10 marks***)

4.4 A buffer is prepared by mixing 50.00 mL of 0.0500 *mol* $dm^{-3}$ ammonia ($K_b = 1.8 \times 10^{-5}$), 30.00 mL of 0.0500 *mol* $dm^{-3}$ HCl and 20.00 mL of distilled water.

4.4.1 Calculate the pH of this solution. (***20 marks***)

4.4.2 If the above buffer solution is diluted to 250.00 mL in a volumetric flask, predict what changes would be in pH of the new solution? (***05 marks***)

@@@@@@@@@@@@@