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UNIVERSITY OF RUHUNA – FACULTY OF MEDICINE
ALLIED HEALTH SCIENCES DEGREE PROGRAMME
THIRD BPHARM PART I EXAMINATION – DECEMBER 2013
PH 3113 ANALYTICAL CHEMISTRY

TIME: TWO (02) HOURS

INSTRUCTIONS

- Answer **all four (04)** questions in the given answer book.
- Use of calculators is allowed.
- No paper should be removed from the examination hall.
- Marks will be penalized for illegible hand writing.
- Do not use any correction fluid.

01. Answer **both** parts

1.1

1.1.1 What is method of validation?

(10 marks)

1.1.2 List **five (05)** parameters of method validation.

(10 marks)

1.1.3 Explain briefly **three (03)** of the parameters listed above in part 1.1.2

(30 marks)

1.2

1.2.1 List **five (05)** sources of impurities in pharmaceutical chemicals.

(10 marks)

1.2.2 Explain briefly **two (02)** of the sources listed above in part 1.2.1

(40 marks)

02 Answer **all** parts

2.1

2.1.1 Define the terms *molarity* and *molality* which are used to express the concentration of a solution?

(04 marks)

2.1.2 A solution of NaCl (M.W. 58.5 g/mol) is prepared by dissolving 0.2925 g of NaCl in 250.0 cm³ of distilled water. Calculate the concentration of this solution in terms of:

2.1.2.1 mol dm⁻³

(10 marks)

2.1.2.2 in ppm

(08 marks)

2.1.2.3 and in ppb

(04 marks)

2.2

2.2.1 What is the criterion of selecting a buffer to maintain a given pH of a solution?

(04 marks)

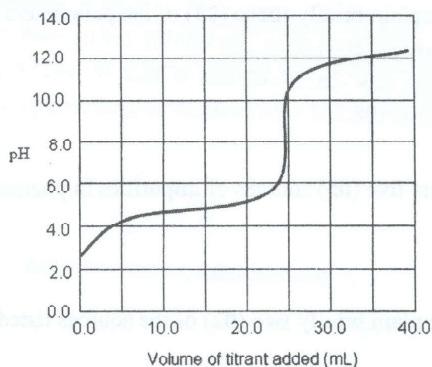
2.2.2 Define the term buffer capacity?

(04 marks)

2.2.3 Calculate the pH of a solution that results when 40.00 cm³ of 0.0500 mol dm⁻³ NH₃ solution is mixed with 20.00 cm³ of 0.0250 mol dm⁻³ HCl solution. (K_b of NH₃ is 1.8x10⁻⁵ at 25 °C)

(16 marks)

2.3 Consider the following titration curve:



2.3.1 Which type of titration does this titration curve represent?

2.3.2 What is the value of the pK_a that can be obtained from this titration curve?

2.3.3 What is the pH of the solution at the point of maximum buffering?

2.3.4 What is the pH of the solution at the equivalence point?

2.3.5 Which of the following indicators would be the most suitable for this titration? phenolphthalein (pK_a = 9.6), cresol red (pK_a = 8.3), methyl red (pK_a = 5.1), methyl yellow (pK_a = 3.1), or any acid/base indicator.

(50 marks)

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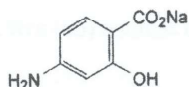
03 Answer **all** parts

- 3.1 What is "KHP"? Draw its chemical structure and explain why it is a good primary standard." (15 marks)
- 3.2 In a typical acid-base titration, a student dissolved 5.2645 g of dry KHP (FW = 204.22) in small amount of deionized water and then diluted to the mark in a 500-mL volumetric flask. He then carefully transferred 100.00 mL of this solution to a clean flask and titrated with 26.75 mL of a Ba(OH)₂ solution to the phenolphthalein endpoint. Calculate the molarity of the Ba(OH)₂ solution. (20 marks)
- 3.3 Suppose that you need to prepare 1.00 L of a Fe₂(SO₄)₃ (FW = 400.0) solution that has a pFe value of 2.50. Determine the mass of Fe₂(SO₄)₃ that you would need and briefly describe how you would prepare the solution. (15 marks)
- 3.4 A third year pharmacy student was asked to carry out a diazotization titration to determine the assay of an antituberculous drug, sodium aminosalicylate (C₇H₆NNaO₃). Given below are the results obtained for this titration:

| Sample | Weight of the sample (g) | Volume of 0.1 M NaNO ₂ required (mL) |
|--------|--------------------------|-------------------------------------------------|
| 1 | 0.5000 | 24.80 |
| 2 | 0.5000 | 25.10 |
| 3 | 0.5000 | 25.00 |

Answer the following questions.

- 3.4.1 Write a balanced chemical equation for the diazotization titration of sodium aminosalicylate.



(10 marks)

- 3.4.2 Calculate the assay of sodium aminosalicylate of the sample. (1 mL of 0.1 M sodium nitrite is equivalent to 17.52 mg of C₇H₆NNaO₃).

(20 marks)

- 3.5 What do you mean by an amphiprotic species? Using H₂PO₄⁻ as the example, explain briefly your answer.

(20 marks)

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04 Answer **all** parts

4.1 A 50.00 mL sample containing Ni(II) was treated with 25.00 mL of $0.0500 \text{ mol dm}^{-3}$ EDTA solution and then back titrated with $0.0500 \text{ mol dm}^{-3}$ standard Zn(II) solution at pH 5.5. The EBT end point was reached at 5.00 mL Zn(II) solution.

4.1.1 What is the concentration of Ni(II) in the sample?

4.1.2 Write the reaction and equilibrium constant expression for the Zn(II) titration.

4.1.3 What is the K'_f value for the Zn complex? $\log K_f$ for the EDTA zinc complex and α_{Y4-} (@ pH 5.5) are 16.50 and 2.3×10^{-5} respectively.

4.1.4 Give one reason why back titrations are used as a method in EDTA Titrations

(60 marks)

4.2 Each of the following substances is used in precipitation titrations. For each substance, state the method in which it is used and explain its specific function. Use appropriate chemical reactions in your answers whenever possible

4.2.1 $\text{Fe}(\text{NO}_3)_3$

4.2.2 potassium thiocyanate

4.2.3 dichlorofluorescein

(20 marks)

(a) In a routine Volhard analysis, a student dissolved a 1.2319-g sample of NaBr in 100 mL of deionized water and then added 100.00 mL of 0.1045 M AgNO_3 . The mixture was then back titrated with 0.1096 M KSCN and 28.65 mL was required to reach the endpoint. Calculate the mass percentage of NaBr (FM = 102.89) in the sample.

(20 marks)

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