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UNIVERSITY OF RUHUNA- FACULTY OF ALLIED HEALTH SCIENCES

DEPARTMENT OF PHARMACY

THIRD BPHARM PART I EXAMINATION – OCTOBER/NOVEMBER 2019

PH 3113 ANALYTICAL CHEMISTRY (SEQ)

TIME: THREE HOURS

INSTRUCTIONS

- There are **four** (04) questions in part A, B and C of the SEQ paper.
- Answer **each part** in a separate booklet provided.
- Use of calculators are allowed
- Do not use any correction fluid.
- Marks will be deducted for illegible hand writing.

PART A

01. Answer all parts.

1.1. What is meant by a "Reference Standard"? (*10 marks*)

1.2. Give **four** uses of Reference Standards. (*20 marks*)

1.3. Define the term 'Purity' pertaining to pharmaceutical materials. (*10 marks*)

1.4. A quality assurance department informed that the aspirin manufactured was not pure. Analysis of a 121.2 g of solid obtained showed that only 109.2 g of it was aspirin. Calculate the percent purity of the product. (*20 marks*)

1.5. State the purpose of good laboratory practices. (*20 marks*)

1.6. List types of analytical procedures that need to be validated. (*20 marks*)

PART B

02. Answer all parts.

2.1

2.1.1 What is a buffer solution? (*05 marks*)

2.1.2 Define the term "buffer capacity". (*05 marks*)

2.1.3 Assume that 0.1 *mol* of H_3O^+ is added separately into two buffer solutions with $[A^-]/[HA]$ ratio equals to 1 and 100. With the aid of a suitable calculation, show that the buffer capacity is high when $[A^-]/[HA]$ ratio equals to 1. (*15 marks*)

2.2. A buffer solution was prepared by adding 500.0 cm^3 of 0.050 *mol* dm^{-3} acetic acid (K_a = 1.75×10^{-5} at 25 °C) and 500.0 cm^3 of 0.050 *mol* dm^{-3} sodium acetate.

2.2.1 Derive the Henderson Hasselbalch equation considering the dissociation of a weak acid (HA). (*05 marks*)

2.2.2 Calculate the pH of the above buffer solution. (*10 marks*)

2.2.3 Calculate the new pH of above buffer solution, if 0.020 *mol* of HCl is added to the above solution. (*Consider that the added volume of HCl is negligible*). (*10 marks*)

PART C

2.3. A solution containing 25.00 mL of ~5 *mol dm*$^{-3}$ HCl was titrated with a KHP solution after adding 50.00 mL of 3.267 *mol dm*$^{-3}$ NaOH. The titration required 23.24 mL of the KHP solution (FW=204.221) which was prepared by dissolving 0.8567 g of KHP in 40.00 mL of H_2O.

2.3.1. Draw the structure of KHP and give the detailed name of it.

2.3.2. Calculate the molarity of the above KHP solution.

2.3.3. Calculate the number of moles of NaOH added.

2.3.4. Calculate the concentration of HCl in the initial solution.

2.3.5. Sketch the titration curve for the above titration and indicate the equivalence point pH.

(50 marks)

03. Answer all parts.

3.1 Following are some stability constants for the formation of metal-EDTA complexes:

Metal ion	K_{MY}	Metal ion	K_{MY}
Mg^{2+}	4.9×10^{8}	Ni^{2+}	4.2×10^{18}
Ba^{2+}	5.8×10^{7}	Cu^{2+}	6.3×10^{18}
Ca^{2+}	5.0×10^{10}	Zn^{2+}	3.2×10^{16}
Mn^{2+}	6.2×10^{13}	Pb^{2+}	1.1×10^{18}

3.1.1 What reaction will occur if a solution containing Pb^{2+} is added to a solution containing MgY^{2-}?

(15 marks)

3.1.2 What reaction will occur if a solution containing Ba^{2+} is added to a solution containing CuY^{2-}?

(15 marks)

3.1.3 Show how a solution containing both Ca^{2+} and Ni^{2+} can be selectively analyzed by buffering to a pH of 3 and 9 in a titration with EDTA whose α_{Y4-} values are 2.6×10^{-11} and 5.4×10^{-9} at pH 3 and 9 respectively.

(30 marks)

3.2 A 25.00 ml aliquot of a solution containing Cu^{2+} and Fe^{3+} was titrated with 16.06 ml of 0.0508 *mol dm*$^{-3}$ EDTA. A second 25.00 ml aliquot of the Cu/Fe mixture was treated with NaF to form a stable iron-fluoride complex. This mixture was then titrated with 0.0508 *mol dm*$^{-3}$ EDTA and the endpoint volume was found to be 5.43 ml. Calculate the molar concentrations of Cu^{2+} and Fe^{3+} in the solution.

(40 marks)

04. Answer all parts

4.1 Write balanced chemical equation and the end point reaction for each of the following titration.

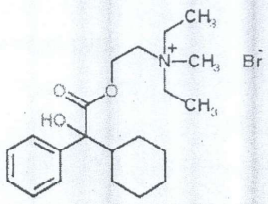
4.1.1 Volhard determination of oxyphenonium bromide (**A,** anticholinergic agent).

4.1.2 Diazotization titration of dapsone (**B,** antibiotic).

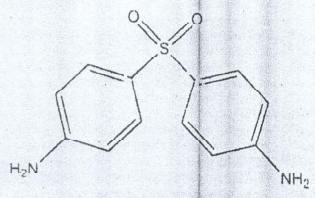
4.1.3 Aceteous perchloric acid titration of adrenaline (**C,** hormone).

4.1.4 Aceteous perchloric acid titration of lidocaine hydrochloride (**D**,local anesthetic).

(***60 marks***)



A



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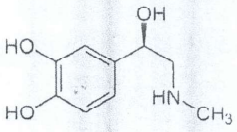
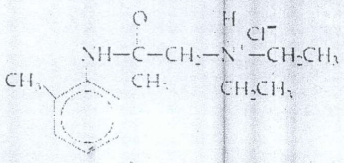


C



D

4.2 Consider the titration of Fe^{2+} and Ce^{4+} and calculate the potential of the indicator electrode at 50%, 100% and 200% of titration, given the following half reactions:

$Fe^{3+} + e \rightarrow Fe^{2+}$ $\quad E^{\circ} = 0.771$ V

$Ce^{4+} + e \rightarrow Ce^{3+}$ $\quad E^{\circ} = 1.44$ V (in H_2SO_4)

(***40 marks***)