PRACTICAL PROBLEMS

PROBLEM 1 (Practical)

Identification of Unknown Solutions

You are supplied with five different solutions contained in five test tubes labelled as A, B, C, D and E, respectively. The solution in each test tube contains one of the following compounds:

NH_4Cl $(NH_4)_2SO_4$ $NaNO_3$ $MgCl_2$ Na_2SO_4

Identify these solutions.

Reagents

H_2SO_4 (conc.)	H_2SO_4 (6 mol dm^{-3})
HNO_3 (conc.)	HNO_3 (6 mol dm^{-3})
HCl (conc.)	HCl (6 mol dm^{-3})
$Ba(OH)_2$ (satd.)	NaOH (6 mol dm^{-3})
$BaCl_2$ (0.5 mol dm^{-3})	$Ba(NO_3)_2$ (0.5 mol dm^{-3})

NOTES:

(1) You can only select the provided reagents and use a procedure as simple as possible to complete your task. You are getting a mark not only according to the correct identification, but also to the number of steps you have taken.

(2) You have to carry out the whole analysis by using the provided amount of these unknown solutions. Supplement of them will be available, but it will reduce the mark you obtain.

PROBLEM 2 (Practical)

Preparation of cis-Copper-bis-Glycinate Hydrate [$Cu(gly)_2 \cdot xH_2O$]

Copper(II) amino acidate coordination compounds are monometric units for synthesizing important biopolymers such as metalloenzymes like ceruloplasmin, on which every living organism depends. In laboratory cis-copper-bis-glycinate hydrate can be produced by the reaction of cupric hydroxide with glycine at a temperature of ca. 70 ℃.

Reagents:

$CuSO_4 \cdot 5\ H_2O$(s)

NH_3 (aq) (3 mol dm^{-3})

glycine(s)

95% ethanol,

acetone

NaOH (2 mol dm^{-3})

$BaCl_2$ (0.5 mol dm^{-3})

1. Preparation of $Cu(OH)_2$

Procedure:

(1) Dissolve your pre-weighted sample of $CuSO_4 \cdot 5\ H_2O$ (6.0 g) in 40 cm^3 of water with a 250 cm^3 beaker as a container.

(2) Add slowly 3 mol dm^{-3} ammonia solution to the $CuSO_4$ solution, gently stirring, until the precipitate is completely dissolved and the solution is turning blue-violet.

(3) Add 2 mol dm^{-3} NaOH solution to the above solution until no more precipitate formed.

(4) Filter the precipitate over a Büchner funnel under reduced pressure. Wash the precipitate with water until no SO_4^{2-} ion is detected in the filtrate.

(5) Collect $Cu(OH)_2$ for the preparation of $Cu(gly)_2 \cdot x\ H_2O$.

Write the equations for the main chemical reactions having taken place in the above procedure.

2. Preparation of cis-Copper-bis-Glycinate Hydrate

Procedure:

(1) Dissolve a pre-weighted sample of glycine (3.6 g) in 130 cm^3 of water and then warm the solution in a hot water bath (70 ℃). Add the $Cu(OH)_2$ to the solution, stir gently until the precipitate is dissolved. Perform a hot filtration and add 10 cm^3 of 95 % ethanol.

(2) Cool the solution and when needle-like crystals appear, place it in the ice water bath for 10 min.

(3) Filter the crystals over a Büchner funnel under reduced pressure, wash once with 10 cm^3 of ethanol-water mixing solvent and then twice with 10 cm^3 acetone, squeeze the crystals as dry as possible on the funnel.

(4) Collect the crystals to a watch glass and dry it (consult your supervisor).

(5) Half an hour later weigh the product. Write the mass of product and the percentage of yield on your student's report. Give the expressions for calculation to show how you calculate.

SOLUTION

The following values were required to be written on the Answer Sheet :

- Mass of the product.
- The calculated theoretical yield in g.
- The yield obtained as a percentage of the theoretical yield.

Tasks:

Write down the balanced chemical equations used in the preparation.

Solution of the tasks:

1. $Cu^{2+} + 2\ NH_3 + 2\ H_2O \rightarrow Cu(OH)_2 + 2\ NH_4^+$

   $Cu(OH)_2 + 4\ NH_3 \rightarrow [Cu(NH_3)_4]^{2+} + 2\ OH^-$

2. $Cu(OH)_2 + 2\ NH_2CH_2COOH \rightarrow Cu(NH_2CH_2COO^-)_2 + 2\ H_2O$

PROBLEM 3 (Practical)

Determination of Copper(II) Content in $Cu(gly)_2 . x H_2O$

The Cu(II) content in $Cu(gly)_2 . x H_2O$ crystals prepared yourself can be determined by iodometry with starch solution as indicator. Based on the data obtained one can calculate

the moles of hydrate in $Cu(gly)_2 . x H_2O$.

Reagents:

Standard KIO_3 (see the label on the bottle to get the accurate concentration)

H_2SO_4 (1.0 mol dm^{-3}) as indicator.

KI (0.6 mol dm^{-3})

KSCN (2 mol dm^{-3})

Starch (0.5 %)

$Na_2S_2O_3$ (to be standardized)

1. Standardization of $Na_2S_2O_3$ solution

Procedure

(1) Transfer 25.00 cm^3 of standard KIO_3 solution to an Erlenmeyer flask.

(2) Add 5 cm^3 of water, 10 cm^3 of KI solution and 5 cm^3 of H_2SO_4 (1.0 mol dm^{-3}) to the flask.

(3) Titrate immediately with $Na_2S_2O_3$ solution.

(4) Add 2 cm^3 starch solution when the colour of the titrand turns pale yellow.

(5) Continue titrating until the blue colour of the solution disappears.

(6) Proceed with step (1) – (5) twice parallel.

2. Determination of Cu(II) content in $Cu(gly)_2.xH_2O$

(1) Weigh 1.0 – 1.2 g (precision of ±0.0002 g) of $Cu(gly)_2 . x H_2O$ with a dry 100 cm^3 beaker as the container.

(2) Dissolve it with 40 cm^3 of water and 8 cm^3 of H_2SO_4 (1.0 mol dm^{-3}).

(3) Transfer the above solution quantitatively to a 100 cm^3 volumetric flask and dilute to the mark.

(4) Transfer 25.00 cm^3 of the Cu(II) solution to an Erlenmeyer flask, add 50 cm^3 of water and 10 cm^3 of KI solution to the flask.

(5) Titrate immediately with standardized $Na_2S_2O_3$ solution.

(6) Add 2 cm^3 of starch solution and 3 cm^3 of KSCN solution to the flask when the colour of the titrand turns from brown to pale yellow.

(7) Titrate continuously until the blue colour of the solution disappears.

(8) Proceed with steps (4) – (7) twice parallel.

SOLUTION

The following values were required to be written on the Answer Sheet :

Part 1

- Volumes of $Na_2S_2O_3$ solution
- Calculation of the concentration of $Na_2S_2O_3$ solution.

Part 2

- Mass of the product.
- Volumes of $Na_2S_2O_3$ solution
- Mass % of Cu(II) in $Cu(gly^-)_2 \cdot x\, H_2O$. Calculation.
- The value of x in the formula of the product. Calculation.

Other Tasks:

1. Write two equations for chemical reactions taking place during the standardization of $Na_2S_2O_3$ solution.
2. Write the equation for the reaction between Cu^{2+} and I^-.

Solutions of the tasks:

1. $IO_3^- + 5\, I^- + 6\, H_3O^+ \rightarrow I_2 + 9\, H_2O$

   $I_2 + 2\, S_2O_3^{2-} \rightarrow 2\, I^- + S_4O_6^{2-}$

2. $2\, Cu^{2+} + 4\, I^- \rightarrow 2\, CuI + I_2$