CHEM 102, CHEMISTRY SESSIONAL

EXPERIMENTAL PROCEDURE

EXPT NO: 06 **DATE: ----------**

NAME OF THE EXPT: ESTIMATION OF COPPER (Cu) CONTENT IN A COPPER SULPHATE PENTAHYDRATE ($CuSO_4.5H_2O$) SOLUTION BY IODOMETRIC METHOD WITH STANDARD SODIUM THIOSULPHATE PENTAHYDRATE ($Na_2S_2O_3.5H_2O$) SOLUTION

Iodometry is an indirect titrimetric analysis of a solution of an oxidizing agent by adding iodide salts which react to form iodine and then this liberated iodine is titrated against standard thiosulphate solution. The amount of iodine liberated from the iodide ion by reacting with an oxidizing agent is the measure of the quantity of the oxidizing agent. Iodometric methods can be used to quantitatively determine strong oxidizing agents such as potassium dichromate, permanganate, hydrogen peroxide, cupric ion and oxygen.

In this experiment, copper (II) sulphate pentahydrate also known as blue vitriol, works as an oxidizing agent and it reacts with excess potassium iodide to liberate iodine which is determined by standard thiosulphate solution. Since the solubility of I_2 is quite low, the formation of tri-iodide ion I_3^-, allows to obtain useful concentrations of I_2 in aqueous solutions. For this reason iodometric titrations are carried out in presence of excess iodide ion. Also, the formation of tri-iodide ion minimizes the loss of iodine by evaporation. The reaction between iodine and the tri-iodide ion can be written as:

$$\mathbf{I_2 + I^- \rightleftharpoons I_3^-}$$

The equilibrium of this reaction is shifted to left when titrated with $Na_2S_2O_3$ or similar reducing agent and more I_2 is liberated from I_3^-. In this manner gradually all the I_3^- decomposes giving off all I_2 during the titration. The reaction of iodine and thiosulphate proceeds quantitatively in neutral or slightly neutral or acidic solutions. In strongly acidic conditions, thiosulphate forms thiosulphuric acid which undergoes an internal oxidation-reduction reaction to sulphurous acid and sulphur and ultimately lead to error since the stoichiometry of the reactions differs from that shown below for the thiosulphate as a reducing agent.

$$\mathbf{2Cu^{2+} + 4I^- = I_2 + 2CuI}$$

$$\mathbf{2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI}$$

To maintain the pH and keep the thiosulphate solution stable, $NaHCO_3$ is used. It is a weak base which helps to maintain pH and slow down the decomposition of thiosulphate.

The end point is sharper when ammonium thiocyanate is added to the solution as it will displace I_2 adsorbed on CuI as CNS^- forms stronger bond than I_2 and makes it available for sodium thiosulphate. Overall oxidation-reduction reaction: $\mathbf{2Cu^{2+} + 2S_2O_3^{2-} + 2I^- \rightarrow 2CuI + S_4O_6^{2-}}$

2.**Apparatus:** Volumetric flask (100ml), Conical flask (100ml), Burette, Pipette (10ml), Funnel, Wash Bottle, Iron Stand etc.

3. **Chemicals:** Copper Sulphate Pentahydrate ($CuSO_4.5H_2O$), Potassium Dichromate ($K_2Cr_2O_7$), Sodium Thiosulphate Pentahydrate ($Na_2S_2O_3.5H_2O$), Potassium Iodide (KI), Sodium bi-carbonate ($NaHCO_3$), Sulfuric acid (H_2SO_4), Ammonium thiocyanate (NH_4SCN), Starch $(C_6(H_2O)_5)_n$

4. **Phase**: The expt can be done into three phases.

A. Prep of standard 0.1 N potassium dichromate ($K_2Cr_2O_7$) solution

B. Standardization of sodium thiosulphate ($Na_2S_2O_3$) solution with standard potassium dichromate ($K_2Cr_2O_7$) solution

C. Determination of copper content iodmetrically with standard $Na_2S_2O_3$ solution

5. **Procedure**:

A. Prep of standard 0.1N Potassium dichromate ($K_2Cr_2O_7$) solution: To prepare 0.1 N 100 mL $K_2Cr_2O_7$ solution, weigh out the required amount (calculate by yourself) of $K_2Cr_2O_7$ and transfer into a 100 mL volumetric flask, dissolve it properly and make up to the mark by adding distilled water.

$$\text{Strength of } K_2Cr_2O_7 \text{ Solution} = \frac{\text{Wt taken}}{\text{Wt to be taken}} \text{ X } 0.1 \text{ N}$$

B. Standardization of $Na_2S_2O_3$ Solution with standard $K_2Cr_2O_7$ Solution:

(i) Take 10 ml of 15 % iodate-free potassium iodide (KI) solution into a conical flask and dilute upto 100 mL with distilled water.

(ii) Add 2 g of sodium bi-carbonate ($NaHCO_3$) and shake until the salts dissolve.

(iii) Add 10 mL of 3N sulphuric acid H_2SO_4 slowly while gently rotating the flask in order to mix the liquids.

(iv) Add 10 mL of standard potassium dichromate ($k_2Cr_2O_7$) solution, mix the solutions well, and wash the sides of the flask with a little distilled water.

(v) Cover the flask with a watch glass and keep it in the dark place for 5 minutes to complete the reaction.

(vi) Rinse the watch glass with distilled water and add it to the conical flask.

(vii) Titrate the liberated iodine with sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution until most of the iodine is reacted as indicated by fading of brown colour or appearance of yellowish-green colour.

(viii) Add 1 mL starch indicator solution with the above solution, intense blue colour appears.

(ix) Continue the addition of the thiosulphate solution dropwise until one drop changes the colour to light green or the blue colour disappears. Repeat the expt three times and note the reading.

(x) Carry out a blank titration, substituting distilled water for the potassium dichromate solution.

Table 1: Standardization of sodium thiosulphate

Ser No	Vol. of $K_2Cr_2O_7$ Solution, V_1 (mL)	Vol. of $Na_2S_2O_3$ Solution				(Mean-Blank), V_2 (mL)	Remarks
		IBR (mL)	FBR (mL)	Diff (mL)	Mean Vol. , V_1 (mL)		
1							
2							
3							
Blank	-				-		

Strength of $Na_2S_2O_3$ **(S_2)** = V_1S_1/V_2

=

Here,

V_1 (Vol of $K_2Cr_2O_7$ solution) =

S_1 (Str of $K_2Cr_2O_7$ solution) =

V_2 (Vol of $Na_2S_2O_3$ Solution) =

S_2 (Str of $Na_2S_2O_3$ solution) = ?

C. Determination of copper content iodmetrically with standard $Na_2S_2O_3$ solution:

(i) Pipette out 10 mL of copper sulphate solution into a conical flask and add about 80 mL of distilled water.

(ii) Add 2 g of sodium-bi-carbonate ($NaHCO_3$) and shake well.

(iii) Add 10 mL 3N sulphuric acid (H_2SO_4) in to the reaction mixture.

(iv) Take 10 mL 12% potassium iodide (KI) solution into the conical flask, cover with watch glass and keep in dark place for 5 minutes to complete the reaction.

(v) Rinse the watch glass with distilled water and add it to the conical flask.

(vi) Titrate with standardized sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution, until the brown colour starts to fade.

(vii) Add 1 mL of starch indicator with the above solution and intense blue colour appear.

(viii) Add 5 mL 10 % ammonium thiocyanate (NH_4CNS) solution.

(ix) Continue the titration until off-white colour appears at the end point.

(x) Carry out a blank titration, substituting distilled water for copper sulphate solution.

Table 2: Determination of Cu^{2+} content

Ser No	Vol of $CuSO_4.5H_2O$ (mL)	Vol. of $Na_2S_2O_3.5H_2O$				Mean- Blank (mL)	Remarks
		IBR(mL)	FBR(mL)	Diff (mL)	Mean Vol. , V_1 (mL)		
1							
2							
3							
Blank	-				-		

10. Calculation for the Estimation of Copper(Cu) content in supplied $CuSO_4.5H_2O$ solution :

1000 ml 1 N $Na_2S_2O_3$ Solution can consume 63.54 g of Cu^{2+}

X ml **Y** N ,, ,, **(63.54 × X × Y) / 1000** g of Cu^{2+}

= **p** **g** of Cu^{2+}.

[Here X and Y are the Vol and Strength of $Na_2S_2O_3$]

10 ml $CuSO_4$ Contains **p** **g** of Cu^{2+}

100 ml ,, ,, **p** × 100 / 10 g of Cu^{2+}

= g of Cu^{2+}

12. **Result:** The Amount of **Copper content** in 100 ml Supplied **$CuSO_4.5H_2O$** Solution is

..... g

13. % of error:

[(Std result ~ Obs result) / Std result] × 100

=

= %

14. Discussion:

KEY POINTS

Difference between iodometric & iodimetric titration, Related half oxidation and reduction reaction, Roll of using $NaHCO_3$ for iodometric titration, Roll of using NH_4SCN in iodometric titration