

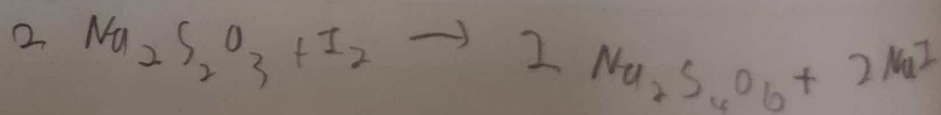
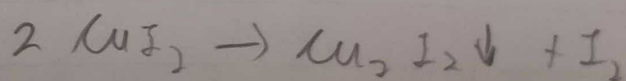
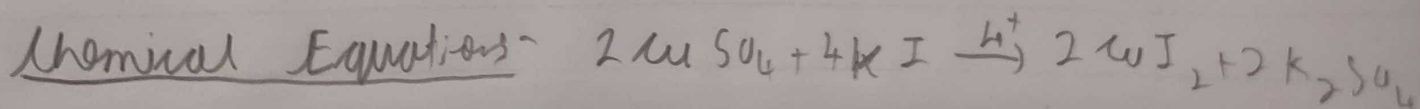
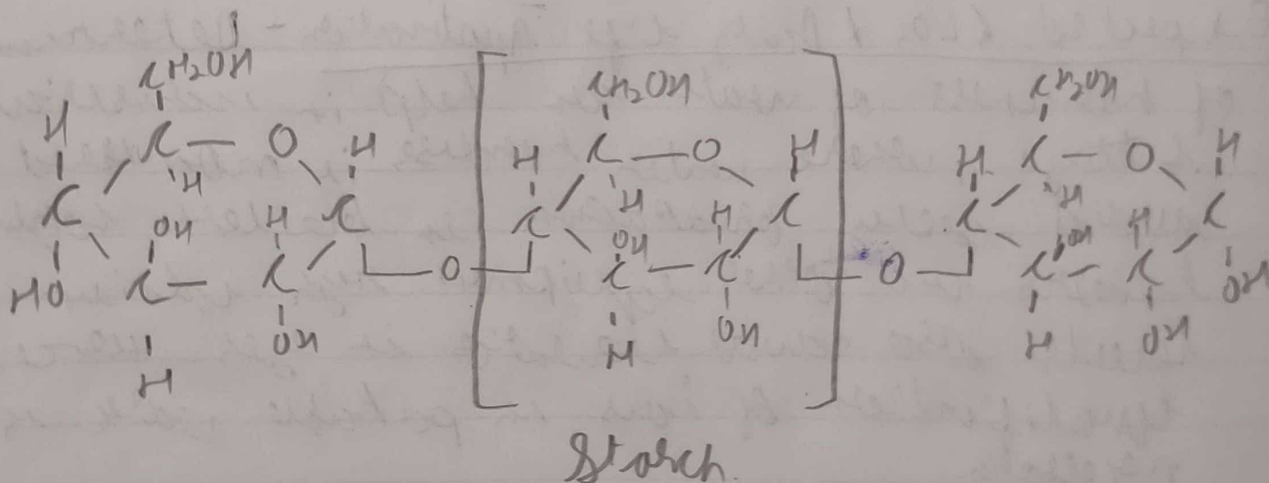
### Experiment- 3

Experiment - To determine the copper content of a given sample of copper ore solution using 0.1 N sodium thiosulphate iodometrically.

Apparatus - Pipette, burette, beakers, conical flask, funnel burette stand and clamp.

Chemicals - Copper sulphate ( $\text{CuSO}_4$ ), sodium bicarbonate ( $\text{NaHCO}_3$ ), acetic acid ( $\text{CH}_3\text{COOH}$ ), Potassium iodide ( $\text{KI}$ ), starch solution and sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ ).

#### Chemical Structure -

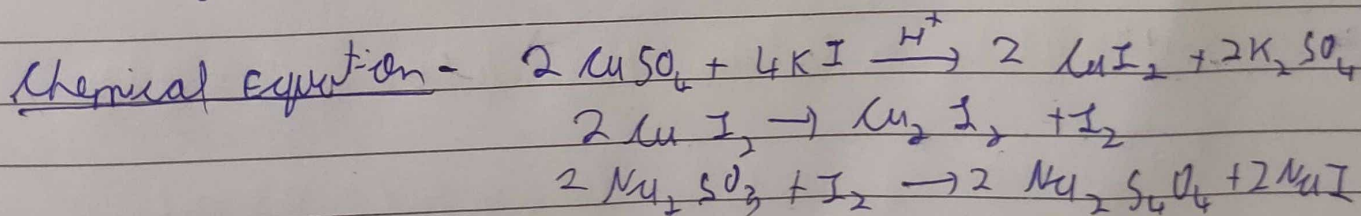


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Chemicals - Copper Sulphate ( $\text{CuSO}_4$ ), Solid sodium bicarbonate ( $\text{NaHCO}_3$ ), acetic acid ( $\text{CH}_3\text{COOH}$ ), potassium iodide ( $\text{KI}$ ), starch solution and sodium thiosulphate.

Theory - Estimation of copper in the copper ore is based on the fact that copper can quantitatively liberate iodide from potassium iodide solution in an acidic medium. The liberated iodine can be titrated against a given standard sodium thiosulphate solution using starch as an indicator.



End point is the appearance of white color due to precipitation of  $\text{Cu}_2\text{I}_2$ . As  $\text{Cu}_2\text{I}_2$  is soluble in mineral acids but insoluble in weak organic acids, the strongly acidic medium is neutralised.

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Indicator - Starch solution.

End point  $\rightarrow$  Starch (pale yellow to deep blue)

Sodium tetrathionate (deep blue to permanent white)

observations - Volume of copper  $\text{or}$  solution taken for each titration = 10 ml

S. No.	Burette Reading (ml)		Volume of 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ sol (ml)
1	0	9.5	9.5
2	0	9.5	9.5
3	0	9.5	9.5
4	0	9.5	9.5

Mean volume of  $\text{Na}_2\text{S}_2\text{O}_3$  used ( $V_2$ ) = 9.5 ml

Calculations - Volume of copper  $\text{or}$  solution used for each titration ( $V_1$ ) = 10 ml

Normality of sodium tetrathionate sol<sup>n</sup> = 0.1 N

Set volume of  $\text{Na}_2\text{S}_2\text{O}_3$  used =  $V_2$  ml = 9.5 ml

using normality eqn

$$N_1 V_1 = N_2 V_2$$

with  $\text{NaHCO}_3$  till a faint permanent precipitate of basic carbonate is formed which is dissolved with a few drops of acetic acid.

Procedure - 1. Pipette out 10 ml of the copper ore sol<sup>n</sup> into a titration flask.

2. Add small amount of same solid  $\text{NaHCO}_3$  to the ore sol<sup>n</sup> in small adds till there is no effervescence the sol<sup>n</sup> turns milky at this stage.
3. Add dilute acetic acid dropwise, just sufficient to remove the milkiness. To the clear blue solution add 5 ml of 10% KI sol<sup>n</sup>. Color of sol<sup>n</sup> changes to dark brown due to the formation of  $\text{KI}_2$ .
4. Add about 35 ml of distilled water to dilute the contents of the flask. Wait for at least 3 min. Titrate the sol<sup>n</sup> against standard sodium thiosulphate sol<sup>n</sup> till the color turns to pale / light yellow.
5. Add about 2 ml of 1% freshly prepared starch sol<sup>n</sup>. Color of the solution turns to deep blue.
6. Continue the titration with more sodium thiosulphate sol<sup>n</sup> till the color changes from blue to permanent white.

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10 ml of  $N_1$  copper ore soln =  $V_2$  ml of 0.1 N  $Na_2S_2O_3$  soln

$$N_1 (\text{Normality of copper soln}) = \frac{0.1 \times V_2}{10}$$
$$= \frac{0.1 \times 9.5}{10} = 0.095 N$$

Equivalent weight of copper = 63.5 g

Amount of copper in the given ore =  $N_1 \times 63.5 \text{ gm/L}$

$$= 0.095 \times 63.5$$

$$= 6.0325 \text{ gm/L}$$

Result - The amount of copper present in copper ore solution is 6.0325 gm/L

7. Keep the contents of the flask for some time on the table shelf. It shouldn't turn blue again. If this happens, add a few more drops of  $\text{Na}_2\text{S}_2\text{O}_3$  sol<sup>n</sup> to get permanent white color again.
8. Repeat the experiment to get at least five correct readings till atleast two concordant readings are obtained.

General calculations - Volume of copper ore sol<sup>n</sup> used for each titration ( $V_1$ ) = 10 ml

Normality of  $\text{Na}_2\text{S}_2\text{O}_3 = 0.1 \text{ N}$

Set volume of  $\text{Na}_2\text{S}_2\text{O}_3$  used =  $V_2$  ml

Applying normality eq. = (copper ore)  $N_1 V_1 = N_2 V_2$  ( $\text{Na}_2\text{S}_2\text{O}_3$ )

10 ml of  $N_1$  copper ore sol =  $V_2$  ml of 0.1  $\text{Na}_2\text{S}_2\text{O}_3$

$$N_1 = \frac{0.1 \times V_2}{10}$$

Amount of copper in given ore =  $N_1 \times 63.5 \text{ gm/L}$

Result - In amount of the copper present in copper ore sol<sup>n</sup> is 6.0325 gm/L

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Precautions - 1. The colour change at the end point should be permanent.

2. The copper ore sol<sup>n</sup> should be neutralised before titration.

3. The contents of the titration flask should be disturbed to observe better change of colour at the end point.

4. After mixing the initial sol<sup>n</sup>, wait for at least 3 min before starting the titration.

5. General precaution of volumetric titrations should be followed.

Daily life Applications - As both excess and lower levels of copper can have adverse effect, eg. excess of copper in body can cause "Wilson Disease".

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