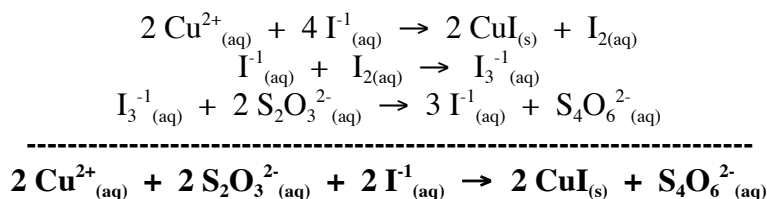


## TITRATION OF WATER SOLUBLE COPPER SALTS

The determination of the percentage copper in an unknown salt can be determined by titration analysis. Titration involves delivering a measured amount of a solution whose concentration is known accurately (the titrant) into a solution whose concentration is not known (the titrate). The purpose is to determine the number of moles present of the reacting species whose concentration is not known. When the reaction is complete, some physical change is observed, indicating the *endpoint* of the titration. The endpoint occurs when stoichiometric equal ratios of reactants are present and must be determined accurately.

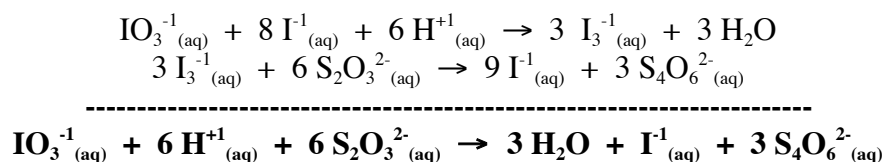
The unknown copper salt acts as part of the titrate solution. The titration involves several simultaneous reactions in solution: i) the copper(II) cation will react with  $I^-$ ; ii) the resulting  $I_2$  can react with excess  $I^-$  to form  $I_3^-$ ; iii) the  $I_3^-$  can be titrated with sodium thiosulfate:



In the present case, a solution of  $\text{S}_2\text{O}_3^{2-}$ , with a known concentration, is acting as the titrant and is delivered into the solution containing  $\text{I}_3^{-1}$  ions, the titrate. When the reaction is complete, all the  $\text{I}_3^{-1}$  anion will be depleted. The endpoint can be detected by adding starch as an indicator;  $\text{I}_3^{-1}$  complexes with starch to form a bright blue color. When the  $\text{I}_3^{-1}$  is depleted, the starch complex is no longer present, and the solution changes from bright blue to colorless.

At this point, the number of moles of  $\text{I}_3^{-1}$  is just sufficient for complete reaction with the  $\text{S}_2\text{O}_3^{2-}$ . The number of moles  $\text{S}_2\text{O}_3^{2-}$  can be determined from its known concentration and volume added. This is then stoichiometrically related to moles of  $\text{Cu}^{2+}$  in the original copper salt. If the mass of the copper salt is known, the % copper in the salt can be determined.

First you must determine the exact concentration of the titrant; the  $\text{S}_2\text{O}_3^{2-}$ . This is done by means of titration with a *standard* solution, a solution whose concentration is known very accurately. We will use potassium iodate as a *primary standard*. A primary standard is a compound whose mass can be determined accurately and which is extremely stable. Sodium thiosulfate cannot be used as a primary standard because it is hygroscopic, and thus not stable over time. The iodate anion reacts with  $I^-$  (a catalyst) to produce  $\text{I}_3^-$  that can be titrated with  $\text{S}_2\text{O}_3^{2-}$ :



**PROCEDURE: Part A: Preparation of Sodium thiosulfate Solution**

Calculate the mass of  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$  needed to prepare 300 mL of approximately 0.1 M solution. Prepare this solution and store in a stoppered Erlenmeyer Flask. Clean a buret with soap and water. Rinse the buret with 5 mL of your thiosulfate solution. This is the only solution you will use in the buret.

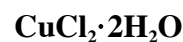
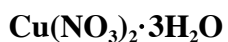
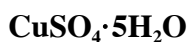
**Part B: Standardization of Thiosulfate Solution**

- Use an analytical balance to weigh two samples of  $\text{KIO}_3$  between 0.1000 and 0.1200 grams.
- Dissolve one sample of  $\text{KIO}_3$  in about 25 mL of water in an Erlenmeyer flask and add about 2 grams of KI.
- Add 19 mL of 1 M HCl and titrate immediately with thiosulfate. Add 5.0 mL of the thiosulfate titrant, then add 5 mL of starch indicator solution. Continue to titrate slowly with thiosulfate until the blue/black color disappears. Record the total volume of thiosulfate added which should be between 25-40 mL.
- Repeat the titration with the second sample of  $\text{KIO}_3$ .
- Calculate the molarity of the thiosulfate solution from the number of grams of  $\text{KIO}_3$  added. Find parts per thousand (ppt).

**Part C: Determination of Unknown Copper**

- Choose an unknown copper salt and record its letter and color. Weigh two roughly 0.3000 gram samples of your unknown copper salt on an analytical balance.
- Just before beginning the titration, weigh about 3 grams of KI and place it in an Erlenmeyer flask. Dissolve the KI in about 25 mL of water and add 19 mL of 0.1 M acetic acid.
- Dissolve one copper salt sample in 20 mL of water in a beaker. Add the copper solution to the acidic KI solution. Rinse the beaker with deionized water to insure that all the copper salt is transferred.
- Titrate with the previously standardized thiosulfate solution. Add 5 mL of thiosulfate, then add 5 mL of starch and complete the titration as before. The end point will be visible when a slightly blue "milkshake" color disappears, leaving a pale pink "milkshake" color behind. Total titrant volume should be about 10 - 25 mL.
- Repeat the titration with a second sample of the same unknown.
- Calculate the average %copper in your unknown salt. Determine parts per thousand.

Your unknown will be one of following copper salts. Determine the **%copper in each salt** as well as the predicted color (only hydrated copper salts have a beautiful blue or green color contrasted to the drab color of unhydrated salts). Record the percentages of all five copper salts in your lab report. Find the **parts per thousand** for the two percent copper values you calculated. Determine the identity of your unknown copper salt from this list based on percentage of copper and color of the original salt.

**POSTLAB QUESTIONS:**

- To standardize a thiosulfate solution, Jean Claude Buret dissolved 0.1250 g of  $\text{KIO}_3$  in HCl and titrates to the endpoint with 23.35 mL of a  $\text{S}_2\text{O}_3^{2-}$  solution of unknown concentration.
  - How many moles of  $\text{KIO}_3$  were present?
  - How many moles of thiosulfate were present at the endpoint of the titration?
  - What was the molarity of the thiosulfate solution?
- Next, Jean Claude's twin, Jeanette, dissolved 0.4550 g of their unknown copper salt in water and added KI as instructed in the lab. She titrated the copper salt solution with 12.45 mL of the above standardized thiosulfate solution to the endpoint.
  - How many moles of thiosulfate were present at the endpoint? How many moles of copper were present?
  - What was the % copper in Jean Claude and Jeanette's unknown copper salt?