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^{-1} ; ii) the resulting I_2 can react with excess I^{-1} to form I_3^{-1} ; iii) the I_3^{-1} can be titrated with sodium thiosulfate:

$$2\ Cu^{2+}_{(aq)} + 4\ I^{-1}_{(aq)} \rightarrow 2\ CuI_{(s)} + I_{2(aq)}$$
$$I^{-1}_{(aq)} + I_{2(aq)} \rightarrow I_3^{-1}{}_{(aq)}$$
$$I_3^{-1}{}_{(aq)} + 2\ S_2O_3^{2-}{}_{(aq)} \rightarrow 3\ I^{-1}_{(aq)} + S_4O_6^{2-}{}_{(aq)}$$

$$\mathbf{2\ Cu^{2+}_{(aq)} + 2\ S_2O_3^{2-}{}_{(aq)} + 2\ I^{-1}_{(aq)} \rightarrow 2\ CuI_{(s)} + S_4O_6^{2-}{}_{(aq)}}$$

In the present case, a solution of $S_2O_3^{2-}$, with a known concentration, is acting as the titrant and is delivered into the solution containing I_3^{-1} ions, the titrate. When the reaction is complete, all the I_3^- anion will be depleted. The endpoint can be detected by adding starch as an indicator; I_3^{-1} complexes with starch to form a bright blue color. When the 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 I_3^{-1} is just sufficient for complete reaction with the $S_2O_3^{2-}$. The number of moles $S_2O_3^{2-}$ can be determined from its known concentration and volume added. This is then stoichiometrically related to moles of 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 $S_2O_3^{2-}$. This is done by means of a 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 I_3^- that can be titrated with $S_2O_3^{2-}$:

$$IO_3^{-1}{}_{(aq)} + 8\ I^{-1}_{(aq)} + 6\ H^{+1}_{(aq)} \rightarrow 3\ I_3^{-1}{}_{(aq)} + 3\ H_2O$$
$$3\ I_3^{-1}{}_{(aq)} + 6\ S_2O_3^{2-}{}_{(aq)} \rightarrow 9\ I^{-1}_{(aq)} + 3\ S_4O_6^{2-}{}_{(aq)}$$

$$\mathbf{IO_3^{-1}{}_{(aq)} + 6\ H^{+1}_{(aq)} + 6\ S_2O_3^{2-}{}_{(aq)} \rightarrow 3\ H_2O + I^{-1}_{(aq)} + 3\ S_4O_6^{2-}{}_{(aq)}}$$

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

Calculate and record the mass of $Na_2S_2O_3 \cdot 5\ H_2O$ needed to prepare 300 mL of approximately 0.1 M solution. Prepare this solution and store in a stoppered Erlenmeyer flask. Rinse a buret thoroughly with water, then rinse the buret with 5 mL of your thiosulfate solution. Fill the buret to 0.00 mL with the thiosulfate solution. This is the ***only*** solution you will use in the buret during this lab.

Part B: ***Standardization of Thiosulfate Solution***

a. Use an analytical balance to weigh two samples of KIO_3 between 0.1000 and 0.1200 grams.
b. Dissolve one sample of KIO_3 in about 25 mL of water in an Erlenmeyer flask and add about 2 grams of KI.
c. Add 19 mL of 1 M HCl to the Erlenmeyer flask 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) to 0.01 mL.
d. Repeat the titration with the second sample of KIO_3.
e. Calculate the **average molarity** of the thiosulfate solution from the number of grams of KIO_3 added. Find **parts per thousand** (ppt). *See the ppt handout in "Lab Notes" of the Companion or website for ppt instructions.*

Part C: ***Determination of Unknown Copper***

a. 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.
b. 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.
c. 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 ensure that all the copper salt is transferred.
d. 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.
e. Repeat the titration with a second sample of the same unknown.
f. Calculate the **average %copper** in your unknown salt. Determine **parts per thousand**.

Your unknown will be one of five copper salts shown below. Determine and record the **percent copper in each copper 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. **Predict** which of these five salts you have in your unknown copper salt.

$CuSO_4 \cdot 5H_2O$ **$CuSO_4$** **$Cu(NO_3)_2 \cdot 2\ ^1/_2\ H_2O$** **$CuBr_2$** **$CuCl_2 \cdot 2H_2O$**

Postlab questions can be found on the next page.

POSTLAB QUESTIONS:

2. To standardize a thiosulfate solution, Jean Claude Buret dissolved 0.1250 g of KIO_3 in HCl and titrates to the endpoint with 23.35 mL of a $S_2O_3^{-2}$ solution of unknown concentration.
   a. How many moles of KIO_3 were present?
   b. How many moles of thiosulfate were present at the endpoint of the titration?
   c. What was the molarity of the thiosulfate solution?

2. 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.
   a. How many moles of thiosulfate were present at the endpoint? How many moles of copper were present?
   b. What was the % copper in Jean Claude and Jeanette's unknown copper salt?

3. **Subscribe** to the *mhchem* email list using your email address of choice (it does not have to be a *saints.mhcc.edu* email address unless you wish it to be so.) Joining is free and easy; complete the form here: **http://mhchem.org/mhchem** For your answer to this problem, **write the email address** you used to subscribe to mhchem.

Lab Notebook and Lab Report: Please read your syllabus for details of lab notebook procedure and lab reports. Information on the Lab Notebook and on how to write a Lab Report can be found here:

http://mhchem.org/lab

Regarding the **Lab Notebook**: While students work in lab groups of 2 or 3 during a lab, each student must have his or her own **lab notebook**. Here are some hints on how to keep a good lab notebook in this class:

- Only use **pen** in your lab notebook; no white out or erasures. If you make a mistake, use a single line to cross out.
- Pages in your lab notebook must be a type that you cannot rip pages out of (composition book) and should be **sequentially numbered** (if they are not, do that now). It is ok to use a lab notebook from a previous course.
- Create a **Table of Contents** at the beginning of your lab notebook (or at the beginning of where you will place your lab information.) The Table should include the title of the lab and the page number at the very least.
- Each lab that you complete should have the **Title** of the lab at the top, followed by your **lab partner or partners** and the **date** that you completed the lab.
- Lab notebooks will be **collected** at some point towards the end of the term for grading.

Lab Reports (which must be typed almost all of the time) are due one week from completion of the experiment unless specified otherwise by the instructor. Each student must write their own lab report. Include all data from the lab report, and show examples of how calculations were made.

Each lab report should include the following:

- your **name**
- your **lab partner(s) name(s) (first AND last)**

- the **title** of the lab
- the **date** that the lab was performed
- a **purpose** section - this is a section with no more than two sentences answering *why* you did this lab.
- a **data** or **results** section - this section should include any relevant data points and calculations obtained within the lab. Make sure you include the **unknown number or letter** if appropriate.
- a **conclusion** section - this section answers the question asked by the purpose
- any **postlab questions**, if any
- In addition, be sure to include any **graphs** or **relevant observations**. If relevant, be sure to include any **averaged values, percent error, parts per thousand (ppt),** etc. All labs must use the correct number of **significant figures** and **units**.

If you have any questions, ask the instructor!