1. Qualitative Analysis of Chromium, Iron, and Copper

Introduction

We have used copper and iron as basic materials since the Bronze and Iron Ages, but our extensive use of chromium began only after the Industrial Revolution. Some of the similarities and differences in the chemical behaviors of the ions of these materials (Ebbing/Gammon, Chapter 13) can become very clear during *qualitative analysis* by chemical methods. Qualitative analysis is the identification of the substances in a mixture. When chemical methods are used in the identification of mixtures of metal cations, these ions are usually separated before identification can occur. After they are separated, identification of each cation depends on the observation of a characteristic chemical reaction.

Purpose

You will learn to separate mixtures of Cr^{3+}, Fe^{3+}, and Cu^{2+} and to identify each ion. Finally, you will be able to determine which of these ions are present in an unknown mixture.

Concept of the experiment

Separation begins when aqueous ammonia is added to a mixture of the three ions. Because aqueous ammonia is a weak base, it is a source of ammonia as well as a source of hydroxide ions. As a consequence, this reagent causes the precipitation of the hydroxides of Cr^{3+} and Fe^{3+}. It also causes the formation of $Cu(NH_3)_4{}^{2+}$, a deep blue complex ion, which remains in solution.

Decanting (pouring off) the solution from the precipitate separates copper from chromium and iron. The presence of copper is confirmed when a red-maroon precipitate of $Cu_2Fe(CN)_6$ forms after the addition of an acid (to neutralize NH_3) and a solution of $K_4Fe(CN)_6$.

To continue the separation, the hydroxides of Cr^{3+} and Fe^{3+} are treated with hydrogen peroxide in basic solution. Only $Cr(OH)_3$ is oxidized. Yellow $CrO_4{}^{2-}$ (chromate ion) is formed in solution, whereas $Fe(OH)_3$ remains as a precipitate. When the solution is decanted from the precipitate, chromium and iron separate. The presence of chromium in the solution is confirmed when a yellow precipitate of $PbCrO_4$ forms after a solution of $Pb(NO_3)_2$ is added.

The precipitate of $Fe(OH)_3$ is then dissolved in an acid. The presence of iron is confirmed by the formation of a deep red color due to $Fe(SCN)^{2+}$, a complex ion, after the addition of a solution of KSCN.

In the Prelaboratory Assignment you will construct a flowchart for the entire scheme in order to become more familiar with it. You can use this scheme to separate and identify any combination of these metal ions in an unknown mixture. Also note that each of these metal ions has a characteristic color. As a result, you should use the color of your unknown mixture to support your analysis using the scheme.

Procedure

Getting started

1. Make sure you are familiar with the flowchart that you completed in the Prelaboratory Assignment.
2. Obtain 6 small test tubes.
3. Obtain your unknown solution and record its identification number and color.
4. Prepare a known mixture of the three ions, using 5 mL of a 0.1 M solution of $Cr(NO_3)_3$, 5 mL of a 0.1 M solution of $Fe(NO_3)_3$, and 5 mL of a 0.1 M solution of $Cu(NO_3)_2$. Note and record the color of each of these solutions before you add it. Make sure that the final solution is thoroughly mixed.
5. Conduct the analysis of the known and the unknown solutions simultaneously so that you can compare the results.
6. Use labeled test tubes so that you do not confuse the known and unknown solutions and precipitates at any time.
7. If necessary, obtain instructions for using the centrifuges in your laboratory.

CAUTION: When you use a centrifuge, do not attempt to stop the centrifuge rotor with your finger or anything else.

8. Obtain directions for discarding the solutions that you will use in this experiment.
9. Take care in handling the solutions used in this experiment.

CAUTION: Ammonia, sodium hydroxide, nitric acid, and acetic acid can cause chemical burns in addition to ruining your clothes. If you spill any of these on you, wash the contaminated area thoroughly with tap water, and report the incident to your instructor. You may require further treatment.

Doing the analysis

1. Take 1 mL of the known mixture and 1 mL of the unknown mixture in separate test tubes.
2. Each of the following additions and operations should be conducted on both mixtures.
3. Add 20 drops of 6 M NH_3 and stir with a clean stirring rod.
4. If no precipitate forms, proceed with Step 12. If a precipitate is present, centrifuge the mixture for about 1 min and decant the solution. Save the solution for Step 12 and use the precipitate in Step 5.
5. Spray the precipitate with about 1 mL of distilled water from a plastic wash bottle. Stir with a clean stirring rod and centrifuge the mixture. Discard the water and use the precipitate in the following step.
6. Add 12 drops of 3% H_2O_2, 1 mL of distilled water, and 5 drops of 6 M NaOH to the precipitate. Let this mixture stand for about 1 min.

7. Light a laboratory burner and heat the mixture gently and cautiously to decompose the remaining H_2O_2. Use a test tube holder. Do not let the flame linger in any one place, or "bumping" will occur. Heat until effervescence from the decomposition ceases.
8. Cool the test tube briefly under tap water. Centrifuge the mixture. Save the solution in a clean test tube for Step 13. If a precipitate remains, use it in Step 9. If no precipitate remains, proceed with Step 13.
9. Wash the precipitate in the same manner as in Step 5.
10. Dissolve the precipitate with 5 drops of 6 *M* HNO_3. Add 3 mL of distilled water, and mix to obtain a homogeneous solution.
11. Add 5 drops of 0.1 *M* KSCN to this solution and mix thoroughly. A deep red color confirms the presence of iron.
12. Make the solution from Step 4 acidic by adding drops of 6 *M* acetic acid until the solution turns blue litmus paper pink. Add 10 drops of 0.1 *M* $K_4Fe(CN)_6$ and mix thoroughly. A red-maroon precipitate confirms the presence of copper.
13. If you did Step 8, make the solution acidic by adding drops of 6 *M* acetic acid until the solution turns blue litmus paper pink. Add 10 drops of 0.1 *M* $Pb(NO_3)_2$ and mix thoroughly. Centrifuge the mixture. A yellow precipitate confirms the presence of chromium.
14. Record the ions that are present in the unknown mixture. Is the color of the unknown mixture in accord with your conclusions? If not, you will need to repeat the analysis.

CAUTION: Before you leave the laboratory, make sure that your gas outlet and those of your neighbors are closed. Finally, wash your hands. Solutions containing lead are poisonous.

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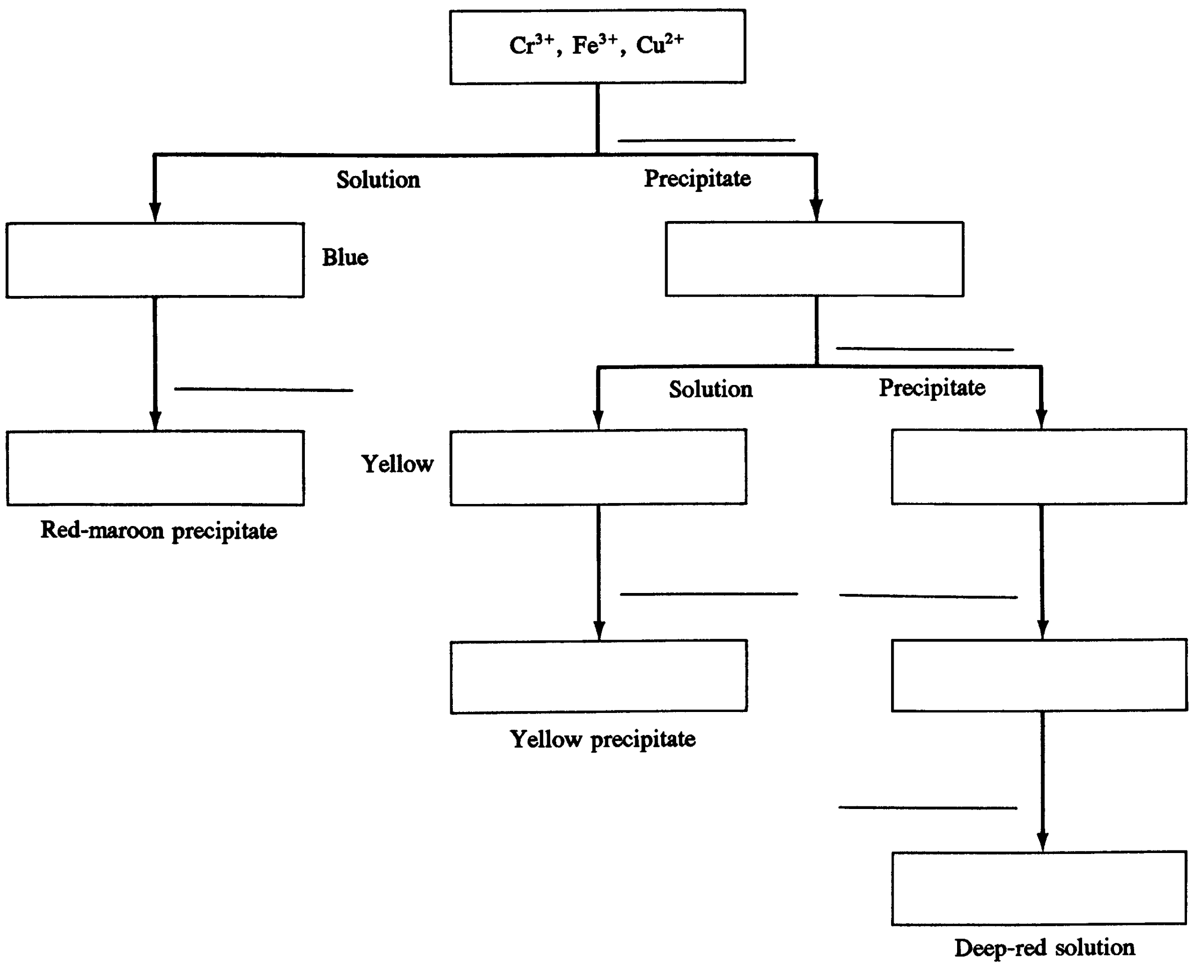
Date:	....	Student name:	....
Course:	....	Team members:	....
Section:	....		....
Instructor:	....		....

Prelaboratory assignment

1. Complete the following flowchart by inserting the reagents and products from each reaction.



2. What special safety precautions are cited in this experiment?

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Results

Unknown no.: ....

Color of unknown mixture: ....

Color of $Cr(NO_3)_3$ solution: ....

Color of $Fe(NO_3)_3$ solution: ....

Color of $Cu(NO_3)_2$ solution: ....

Ions present in unknown mixture: ....

Questions

1. Write a balanced chemical equation for each reaction that appears in the flowchart in the Prelaboratory Assignment.

2. In order to decompose excess hydrogen peroxide during this experiment, you heated the solution until effervescence ceased. The effervescence was due to the evolution of O_2. No other gas was evolved. Write the chemical equation that describes this decomposition.

3. Suppose a solution contained only one cation: Cr^{3+}, Fe^{3+}, or Cu^{2+}. What would you do to identify the cation quickly?