Class

Microscale Tests for Iron(II) and Iron(III)

In this experiment, the complex hexacyanoferrate(II) ion (*ferro*cyanide), $Fe(CN)_6^{4-}$, and the hexacyanoferrate(III) ion (*ferric*yanide), $Fe(CN)_6^{3-}$, will be used in identification tests for Fe^{2+} and Fe^{3+} ions. The charges on the two complex ions clearly indicate the difference in the oxidation state of the iron present in each. The (CN) group in each complex ion has a charge of -1. Thus, iron(II) is present in the *ferro*cyanide ion, $[Fe^{2+}(CN^{-})_6]^{4-}$. Iron(III) is present in the *ferro*cyanide ion, $[Fe^{2+}(CN^{-})_6]^{4-}$. Iron(III) is present in the *ferro*cyanide ion group, $[Fe^{3+}(CN^{-})_6]^{3-}$. A deep blue precipitate results when either complex ion combines with iron in a different oxidation state from that present in the complex. The deep blue color of the precipitate is caused by the presence of iron in both oxidation states. The color provides a means of identifying either iron ion. If the deep blue precipitate is formed on addition of the $[Fe^{2+}(CN^{-})_6]^{4-}$ complex, the iron ion responsible must be the iron(III) ion. Similarly, a deep blue precipitate formed with the $[Fe^{3+}(CN^{-})_6]^{3-}$ complex indicates the presence of the iron(III) ion.

Both of the deep blue precipitates are known to have the same composition. The potassium salt of the complex ion has the formula $KFeFe(CN)_6$ ·H₂O.

The thiocyanate ion, SCN^- , provides a test for confirming the presence of Fe^{3+} ion. The soluble $FeSCN^{2+}$ complex imparts a blood red color to the solution.

OBJECTIVES

Observe tests of known solutions containing iron(II) or iron(III) ions.

Compare results for the two ions and infer conclusions.

Design a procedure for identifying the two ions in one solution.

MATERIALS

- $\rm FeCl_3,\,0.1~M$
- Fe(NH₄)₂(SO₄)₂, 0.2 M
- gloves
- K₃Fe(CN)₆, 0.1 M
- K₄Fe(CN)₆, 0.1 M
- KSCN, 0.2 M

- lab apron
- \bullet plastic wrap, 8 cm \times 30 cm
- safety goggles
- sheet of paper, white
- thin-stemmed pipets (5)

Always wear safety goggles, gloves, and a lab apron to protect your eyes and clothing. If you get a chemical in your eyes, immediately flush the chemical out at the eyewash station while calling to your teacher. Know the location of the emergency lab shower and eyewash station and the procedures for using them.

Tests for Iron(II) and Iron(III) continued

Do not touch any chemicals. If you get a chemical on your skin or clothing, wash the chemical off at the sink while calling to your teacher. Make sure you carefully read the labels and follow the precautions on all containers of chemicals that you use. If there are no precautions stated on the label, ask your teacher what precautions to follow. Do not taste any chemicals or items used in the laboratory. Never return leftover chemicals to their original containers; take only small amounts to avoid wasting supplies.



Call your teacher in the event of a spill. Spills should be cleaned up promptly, according to your teacher's directions.

Acids and bases are corrosive. If an acid or base spills onto your skin or clothing, wash the area immediately with running water. Call your teacher in the event of an acid spill. Acid or base spills should be cleaned up promptly.

Procedure

- **1.** Put on safety goggles, gloves, and a lab apron.
- **2.** Check that you have a labeled pipet for each solution listed in the materials.
- **3.** Place the plastic-wrap rectangle on a white sheet of paper.
- **4.** Along the top of the plastic wrap, place 5 drops of a freshly prepared iron(II) ammonium sulfate solution in each of the three locations shown in Figure 1.
- **5.** Along the bottom of the plastic wrap, place 5 drops of a freshly prepared iron(III) chloride solution, as shown in Figure 1.



Iron(III) chloride

- **6.** Add 1 drop of 0.1 M K_4 Fe(CN)₆ solution to the first sample of iron(II) ions at the top of the plastic wrap and l drop to the first sample of iron(III) ions at the bottom. Record your observations in **Table 1**.
- 7. Add 1 drop of 0.1 M KSCN solution to the second sample of iron(II) ions and 1 drop to the second sample of iron(III) ions. Record your observations in Table 1.

Tests for Iron(II) and Iron(III) continued

- **8.** Add 1 drop of 0.1 M K_3 Fe(CN)₆ solution to the third sample of iron(II) ions and 1 drop to the third sample of iron(III) ions. Record your observations.
- **9.** Clean all apparatus and your lab station. Return equipment to its proper place. Dispose of chemicals and solutions in the containers designated by your teacher. Do not pour any chemicals down the drain or in the trash unless your teacher directs you to do so. Wash your hands thoroughly before you leave the lab and after all work is finished.

TABLE 1: RESULTS OF TESTS FOR IRON(II) AND IRON(III)

Iron ion	Hexacyanoferrate(II) ion $[Fe^{2+} (CN^{-})_6]^{4-}$	Hexacyanoferrate(III) ion [Fe ³⁺ (CN ⁻) ₆] ³⁻	Thiocyanate ion SCN ⁻
Fe ²⁺			
Fe ³⁺			
Observations in step 6			
Observations in step 7			
Observations in step 8			

Analysis

1. Organizing Ideas Explain specifically how you would make a conclusive test for an iron(III) salt.



3. Relating Ideas When iron(II) ammonium sulfate was mixed with the $[Fe^{2+}(CN^{-})_{6}]^{4-}$ ion, the precipitate was initially white but turned blue upon exposure to air. What happened to the iron(II) ion when the precipitate turned blue?

Tests for Iron(II) and Iron(III) continued

Conclusions

- 1. Designing Experiments Suppose you have a solution containing both an iron(II) salt and an iron(III) salt. How would you proceed to identify both Fe^{2+} and Fe^{3+} in this solution?
- **2. Relating Ideas** Blueprint paper can be made by soaking paper in a brown solution of $[Fe^{3+}(CN^{-})_6]^{3-}$ and iron(III) ammonium citrate. Wherever the paper is exposed to bright light, the paper turns blue. Explain why this happens.