

Chromium and Manganese

One of your most important roles as a scientist is making observations while you are performing an experiment. Throughout this course you will be performing reactions and “things” will be happening. By making careful observations you will have important data to help you hypothesize about the nature of the reaction you’ve performed. In this lab you will perform a number of reactions involving the transition metals chromium and manganese. Most transition metal ions can be found in a number of possible oxidation, and can form a variety of complex species. Since we can’t place our molecules under a microscope to see what we have made, we have rely on observations, a knowledge of the reaction conditions, and information available in the literature to help understand the processes that have occurred. In this lab the “literature” will consist of the following tables of ions, their oxidation states, and their colors.

Compound	Color	Oxidation state
Cr(s)	Silver - metallic	0
Cr ²⁺ (aq)	Blue	2
CrCl ⁺ (aq)	Blue-green	2
Cr ³⁺ (aq)	Purple	3
CrCl ₂ ⁺ (aq)	Green	3
Cr(SO ₄) ⁺ (aq)	Green	3
Cr(SO ₃) ⁺ (aq)	Green	3
Cr(OH) ₄ ⁻ (aq)	Green	3
CrO ₄ ²⁻ (aq)	Yellow	6
Cr ₂ O ₇ ²⁻ (aq)	Yellow-orange	6

Compound	Color	Oxidation state
Mn (s)	metallic	0
Mn ²⁺ (aq)	Colorless	2
Mn(OH) ₂ (s)	Off white	2
Mn ₂ O ₃ (s)	Brown	3
MnO ₂ (s)	Black	4
MnO ₄ ²⁻ (aq)	Green	6
MnO ₄ ⁻ (aq)	Purple	7

The color changes during the course of a reaction can tell you a great deal about what has happened. Although several ions may have the same color, because of the reagents present only one will make chemical sense.

For example, suppose a purple solution of $\text{Cr}(\text{NO}_3)_3$ is reacted with hydrochloric acid giving a green solution. From general chemistry you should recall that all nitrate salts are strong electrolytes, that is, they dissociate completely into their substituent ions in water. So the initial solution contained Cr^{3+} ions and nitrate ions (nitrate ion is a complex ion and doesn't dissociate into nitrogen ions and oxygen ions in water!) From Table 1 we can confirm that the initial purple color is from the presence of aqueous Cr^{3+} ions. Hydrochloric acid, being a strong acid, dissociates completely into hydrogen ions and chloride ions in water. The resulting solution is green, which from the table could be one of four species. However since there were no sulfate, sulfite, or hydroxide ions present in the reaction the only conclusion that makes chemical sense is that the green color is due to the production of aqueous CrCl_2^+ from the reaction. Note that this product is a complex cation, just like nitrate ion is a complex anion. It does not dissociate into Cr^{3+} and chloride ions otherwise the color observed would still be purple. You should be able to write a balanced equation for this reaction.

For each reaction that you perform you should record in your notebook

- The color of the solution
- The presence of any solids
- If solid is present does it settle by itself, with centrifugation?
- Color of solids
- Texture of solids (ie granular, curdlike, flocculent, gelatinous)

Experimental Procedure

Chromium reactions

1. In a small test tube place a piece of chromium metal. Add 1 mL of 3 M HNO_3 .
2. pour the 3M nitric acid out of the tube, leaving the remaining metal in the tube. Add 1 mL of conc. nitric acid to the tube.
3. Add a piece of chromium metal to a clean test tube. Add 3 mL of 3 M HCl to the tube. If nothing happens carefully warm the test tube.
4. Take the solution from 3 and split it by carefully pouring it into two clean test tubes. Place a rubber stopper in one tube and shake the solution for 1 minute. Compare with the test tube that wasn't shaken.
5. Stopper and shake the second solution for 1 minute.

6. To one of the two tubes add a small amount of zinc powder and carefully heat until bubbles are observed. Centrifuge the solution.
7. Place a small piece of chromium in a test tube and add 3 mL of 3M sodium hydroxide.
8. Measure 3 mL of 0.1 M $\text{Cr}(\text{NO}_3)_3$ into a small test tube. Add 6 M NaOH drop by drop to the tube. Continue adding sodium hydroxide until no changes are apparent.
9. Using the solution from 8. Add 3% hydrogen peroxide drop-wise, with mixing, to the tube until the color stops changing.
10. Transfer 10 drops of the solution from 9 to a clean test-tube. Add 2.5 mL of water.
11. In a test tube combine 5 ml of water, 1 mL of 1 M K_2CrO_4 and 1 mL of 3 M H_2SO_4 . To this add a few crystals of iron(II)sulfate.
12. Set up a new tube similar to 11. But add a few crystals of Na_2SO_3 .

Manganese.

1. Place a piece of manganese metal in a test tube. Add 5 mL of 3 M HCl.
2. Add 3 mL of 0.1 M $\text{Mn}(\text{NO}_3)_2$ to a test tube. Add 5 drops of 6 M NaOH.
3. Stopper tube 2 and shake for approximately 1 minute.
4. Add 5 mL of water and 5 drops of 0.1 M $\text{Mn}(\text{NO}_3)_2$ to a test tube. Add 1 mL of 6 M HNO_3 . Add a small amount of NaBiO_3 .
5. Mix 3 mL of 0.01 M KMnO_4 and 1 mL of 3 M H_2SO_4 in a test tube. Add a small amount of NaSO_3 .
6. Add 3 mL of 0.01 M KMnO_4 to a test tube. Add 2 drops of 6M NaOH. Add a small amount of Na_2SO_3 . Let the reaction sit for a few minutes then transfer to a centrifuge and centrifuge the solution.
7. In a test tube, mix 3 mL of 0.01 M KMnO_4 and 3 mL of 3M NaOH. Add a small amount of Na_2SO_3 .

Waste:

Zinc(II) Chloride 1%

Chromium(III)chloride 3%

Manganese(II) chloride 3%

Iron(II)sulfate 1%

Sodium sulfite 2%

Sodium bismuthate 1%

Nitric acid 2%

Hydrochloric acid 2%

Sulfuric acid 2%

Water 83%