

The Synthesis of Alum

The term *alum* is a general family name for a crystalline substance composed of cations with 1+ and 3+ charges. In this experiment, you will synthesize a type of alum called potassium aluminum sulfate dodecahydrate, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$. You will synthesize this compound by placing the appropriate ions in one container in aqueous solution and then evaporate the water to form the alum crystals.

This particular compound has been chosen because it is relatively simple to prepare a pure sample. The process of synthesizing this compound is interesting in that it involves both chemical and physical reactions. Chemically, aluminum is oxidized from aluminum foil to prepare the Al^{3+} ions. Physically, as the solution that contains the mixture of ions evaporates, crystals will form which contain six waters of hydration bonded to the aluminum ion and six waters bonded to the potassium ion.

Aluminum is considered a reactive metal, but because its surface is usually protected by a thin film of aluminum oxide, it reacts slowly with acids. It does, however, dissolve quickly in basic solutions. Excess hydroxide ion converts the aluminum to the tetrahydroxoaluminate (III) ion, $[\text{Al}(\text{OH})_4]^-$, and a slow addition of acid will precipitate the white, gelatinous aluminum hydroxide, $\text{Al}(\text{OH})_3$. Continued addition of acid causes the hydroxide ions to be completely neutralized, and the aluminum exists in solution as the hydrated ion $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$. Aluminum hydroxide is considered to be an amphoteric hydroxide because it dissolves in both acids and bases.

OBJECTIVES

In this experiment, you will

- Synthesize a sample of potassium aluminum sulfate dodecahydrate (alum).
- Observe and record the process of synthesizing a compound.
- Calculate the percent yield of your synthesis.

MATERIALS

250 mL beaker
25 mL or 50 mL graduated cylinder
Büchner funnel and filter flask
watch glass
glass stirring rod
lab burner, ring stand, ring, wire gauze
plastic wrap or Parafilm®
fume hood
hot plate

aluminum foil
3 M sulfuric acid solution, H_2SO_4
baking soda, NaHCO_3
3 M potassium hydroxide solution, KOH
aqueous ethanol solution, 50%
vinegar, dilute CH_3COOH solution
ice bath
balance

PROCEDURE

1. Obtain and wear goggles.
2. Obtain a piece of aluminum foil and measure its mass. For best results, you should have about 1.00 g of aluminum. Tear the foil into small pieces and place the pieces in a 250 mL beaker.
3. Set up a Büchner funnel and filter flask so that you are ready to filter the reaction mixture that will be produced in Step 4.
4. Conduct the first part of the synthesis. **CAUTION:** *Potassium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.*
 - a. Use a graduated cylinder to measure out 25 mL of 3 M KOH solution.
 - b. *Slowly* add the KOH solution to the beaker of aluminum pieces. Notice that the reaction is exothermic. Allow the reaction to proceed until all of the foil is dissolved.
 - c. Carefully pour the reaction mixture through your Büchner funnel and filter flask setup, and rinse the filter paper with a small amount of distilled water. **Note:** The reaction mixture contains three ions: K^+ , $[Al(OH)_4]^-$, and excess OH^- .
 - d. Rinse the beaker with distilled water, and pour the filtered liquid back into the beaker.
5. Allow the solution to cool to near room temperature. If you are pressed for time, you may cover the beaker with plastic wrap or Parafilm, and store the liquid overnight.
6. Clean the Büchner funnel and filter flask, and prepare it for more filtering that you may need to do in Step 7 or Step 10.
7. Complete the synthesis.
 - a. Use a graduated cylinder to measure out 35 mL of 3 M H_2SO_4 solution. **CAUTION:** The reaction mixture must be cooled to room temperature before proceeding. Handle the H_2SO_4 solution with care. It can cause painful burns if it comes in contact with the skin.
 - b. After the reaction mixture has cooled, slowly add the sulfuric acid solution to the beaker of liquid. Stir the mixture constantly. The reaction is strongly exothermic, so be careful as you stir the mixture. Note that aluminum hydroxide will precipitate initially. It will dissolve as more sulfuric acid is added.
 - c. If there is some solid remaining in the beaker after the 35 mL of sulfuric acid has been added, pour the mixture through the Büchner funnel and filter flask to separate the undissolved solid from the mixture.
8. Gently boil your mixture until you have about 50 mL of liquid in the beaker.
9. Cool the beaker of solution. Choose one of the two methods listed below.
 - a. Allow the solution to cool overnight. In most cases, this gradual cooling forms a good crop of alum crystals.
 - b. Prepare an ice bath for the 250 mL beaker. Place your beaker of solution, uncovered, in the ice bath. Do not move the ice bath or the beaker. After about fifteen minutes, crystals of alum will appear in the beaker. If there are no crystals after fifteen minutes, scrape the bottom of the beaker with a glass stirring rod to create a rough spot for crystal growth. You may also heat the solution to evaporate more water and cool the solution again.

10. Collect your alum crystals by pouring them onto the Büchner funnel and filter-flask setup. Use vacuum filtration to wash the crystals on the filter paper with 50 mL of an aqueous ethanol solution (50%). The crystals will not dissolve in this solution.
11. Remove the filter and crystals from the Büchner funnel and allow the crystals to dry at room temperature. Measure and record the mass of your sample of alum. Store the crystals for further analysis.

DATA ANALYSIS

1. Determine the theoretical yield of the alum. Use the aluminum foil as the limiting reagent and presume that the foil was pure aluminum.
2. Calculate the percent yield of your alum crystals.
3. Discuss the factors that affected the percent yield.
4. Write the balanced net ionic equations for the following: (a) aluminum and potassium hydroxide, yielding $[\text{Al}(\text{OH})_4]^-$ and hydrogen gas; (b) hydrogen ions and $[\text{Al}(\text{OH})_4]^-$, yielding aluminum hydroxide; (c) aluminum hydroxide and hydrogen ions, yielding $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$; and (d) the formation of alum from potassium ions, sulfate ions, $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$, and water.
5. Describe a synthesis reaction.
6. Describe how the solubility of alum in various solvents and water at different temperatures was used in conducting the experiment.
7. Write a detailed description of the alum crystals.