

## Synthesis of Alum from Aluminum

### OBJECTIVES

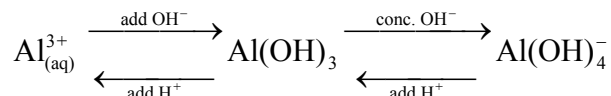
- Become more familiar with single-replacement redox reactions
- Practice mass and volume measurement techniques
- Calculate the theoretical yield and percent yield of a synthesis reaction

### DISCUSSION

**Alums** are ionic compounds that crystallize from solutions containing sulfate ion, a trivalent cation such as  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ , or  $\text{Fe}^{3+}$  and a monovalent cation such as  $\text{K}^+$ ,  $\text{Na}^+$ , or  $\text{NH}_4^+$ . Six of the water molecules bind tightly to the trivalent metal ion; the remaining six molecules bind more loosely to the monovalent cation and the sulfate anion.

In this experiment you will prepare an alum— $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  [potassium aluminum sulfate dodecahydrate]—from scrap aluminum. This compound is widely used in dyeing fabrics, making pickles, making paper and purifying water.

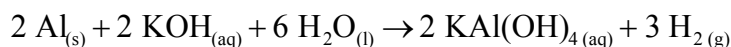
Although aluminum metal sits well above hydrogen in the activity series, it reacts only slowly with dilute acids because a thin coating of aluminum oxide protects the metal surface. Aluminum reacts with alkaline solutions to produce hydrogen because the excess hydroxide ion first attacks the tough  $\text{Al}_2\text{O}_3$  layer so the metal can react. Aluminum converts to the tetrahydroxoaluminate ion  $\text{Al}(\text{OH})_4^-$ . Slow addition of acid to a solution of this ion causes the precipitation of solid  $\text{Al}(\text{OH})_3$  followed by the dissolving of the precipitate to form the aluminum ion  $\text{Al}^{3+}$ . The solid  $\text{Al}(\text{OH})_3$  will also dissolve in excess base due to formation of  $\text{Al}(\text{OH})_4^-$ . We call a hydroxide that can react with either acids or bases *amphoteric*.



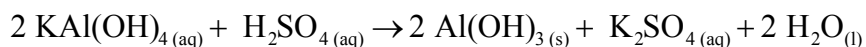
Except for metals in Group 1,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ , and  $\text{Ba}^{2+}$ , all metal ions form insoluble hydroxides. These insoluble hydroxides dissolve in acid solution. Amphoteric hydroxides also dissolve in strong base solutions (concentrated solutions of hydroxide ion made by dissolving solid NaOH or KOH in water).

The equations given below show the complete sequence of reactions involved in our experiment.

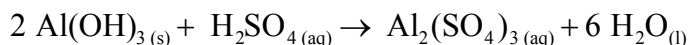
1. Reaction of aluminum with KOH (the dissolution step)



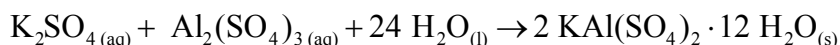
2. Initial addition of sulfuric acid (precipitation of  $\text{Al}(\text{OH})_3$ )



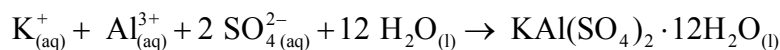
3. Further addition of sulfuric acid (dissolving of  $\text{Al}(\text{OH})_3$ )



4. Precipitation of alum on cooling



It is possible to write an overall net ionic equation for the precipitation process:



## SAFETY

Potassium hydroxide is a strong base. Solutions of it, if splashed into the eye, can do irreparable damage so quickly that it is not possible to reach the eyewash fountain in time to prevent it. **You must wear your goggles throughout this experiment.**

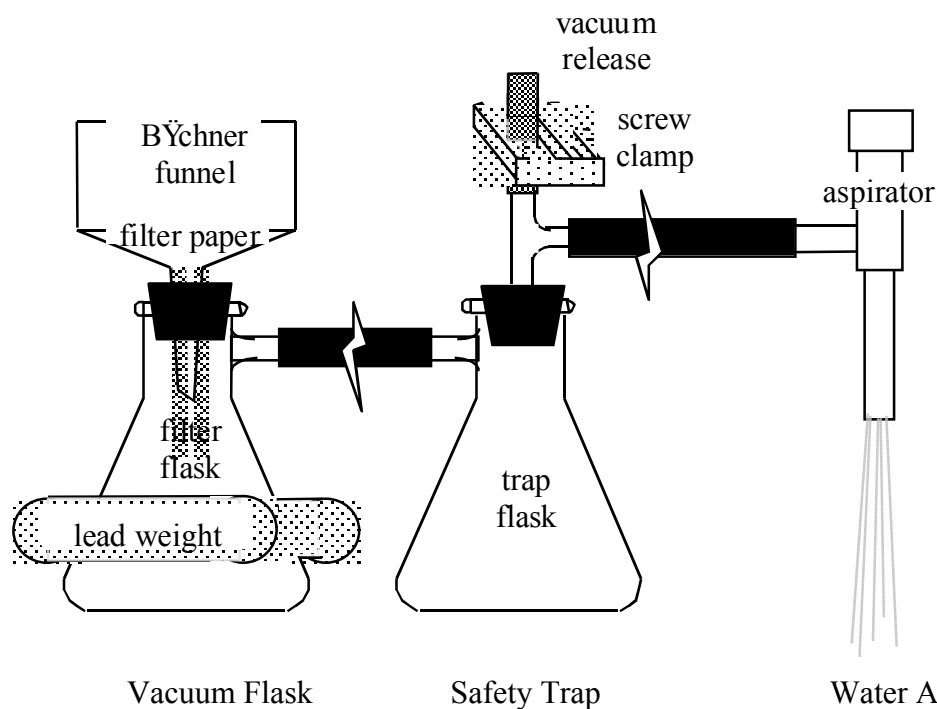
Hydrogen gas is given off when aluminum metal is reacted with potassium hydroxide solutions. Hydrogen is extremely flammable. You should not use your Bunsen burner while you or others are conducting this part of the experiment.

## PROCEDURE

1. Either bring your own aluminum can or use the pieces provided in the lab. You will need a piece of scrap aluminum about 7.5 cm by 5.0 cm that weighs 1.0 to 1.1 grams.
2. Scrape both sides of the piece (using sandpaper) to remove the paint and lacquer (this will speed the dissolving process GREATLY, and give a more accurate starting mass).
3. Weigh the cleaned piece of aluminum on the analytical balance to the nearest milligram.
4. Cut the weighed piece of aluminum into small pieces (for more rapid dissolving) and place the pieces in a 250-mL beaker.
5. **MAKE SURE YOU ARE WEARING YOUR SAFETY GOGGLES!** Working under the fume hood, add 50 mL of 1.4M potassium hydroxide solution to the beaker.
6. Heat the mixture on the electric hot plates provided. If the liquid level drops to half of its original value, add distilled water to restore the original volume. During the reaction, the mixture will turn dark grey due to silicon impurities, residual paint, plastic liners, *etc.* No more than thirty minutes should be required to dissolve the sample. The final liquid volume should be about 60% of the original volume. The reaction is complete when the evolution of hydrogen ceases.
7. Filter the cooled solution to remove any solids remaining. The easiest filtration uses the Büchner funnel and water suction. *Read the separate section on the assembly and use of this device.* Be sure to use the safety trap, because in this first filtration you must save the filtrate (the solution). If you don't use the trap and there is even a small interruption in the water flow, tap water can suck back into your solution and contaminate it.
8. Discard the dark solid trapped in the filter paper and SAVE the solution for use in the next part of the experiment. (Don't waste time trying to recover the small amount of alum that forms in the beaker or suction flask).
9. Transfer the CLEAR (no suspended matter) filtrate to a *clean* 250-mL beaker.
10. If the beaker feels warm, place it in an ice water bath.
11. **BE SURE YOU ARE WEARING YOUR SAFETY GOGGLES.** Slowly and carefully, stir in 20 mL of 9M sulfuric acid. Initially you will notice the formation of a white, gelatinous aluminum hydroxide precipitate. As you add more of the sulfuric acid, you will notice the evolution of heat and the dissolving of the precipitate. If the reaction goes slowly, warm GENTLY while stirring to complete the reaction.

12. Evaporate the solution to a volume of about 60 mL using a hot plate. If any solid remains, filter the mixture by water suction and save the clear filtrate for the next step.
13. Prepare an ice bath by filling a 600-mL beaker half-full with crushed ice and just covering the ice with water. Alternatively, use a small metal pan filled with an ice-water mixture. Set the beaker containing the clear filtrate from the last step into the ice-water mixture to cool. Alum crystals should form after about 15 minutes. Good crystals take time to form, so don't hurry.
14. While the mixture is cooling, clean and reassemble the suction filtration apparatus. When you have a good crop of alum crystals, filter them from the cold solution, transferring as much of the solid product as possible to the funnel.
15. Mix 10 mL of ethanol (ethyl alcohol) and 5 mL of water. Chill this mixture in the ice bath for a few minutes. Using an eyedropper, rinse the remaining crystals from the beaker into the funnel. Rinse the crystals in the funnel with drops of the alcohol-water solution. Allow the crystals to dry for five minutes on the filter paper with full suction.
16. After the crystals are dry, weigh them on the lab balance by the following procedure. Weigh a clean beaker on the balance, then place the crystals in the beaker and weigh the beaker plus crystals.
17. From the mass of aluminum originally recorded, calculate the theoretical yield of alum.
18. From the mass of product recovered and the calculated theoretical yield of alum, calculate your percent yield.
19. Obtain your instructor's approval of your alum crystals.

If your instructor wishes to grow a large crystal, turn in your alum for use as the starting material. If you do not use your alum in this way, dispose of it in the waste container provided.

**Figure 1: Set-up for a suction filtration**

**Vacuum Flask**  
The stockroom staff have already assembled these heavy-walled glass flasks.

**Safety Trap**  
The trap will catch any tap water that kicks back in case of a flow interruption.

**Water Aspirator**  
The partial vacuum created by the rapid flow causes suction.

To break suction when you wish to stop filtering, open the screw clamp on the trap. This will minimize the danger of tap water being drawn back into the filtrate in your vacuum flask. The suction is so strong that the Büchner funnel cannot be lifted out while the system is connected. Once the suction breaks, it can be removed from the vacuum flask. After you have broken the suction, the water faucet can be turned off to stop the flow of water.

Name \_\_\_\_\_ Date \_\_\_\_\_ Score \_\_\_\_\_

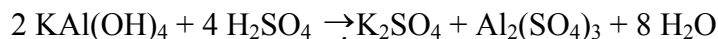
**Prelaboratory Assignment**

1. Calculate the amount in moles and the mass in grams of KOH needed to convert 1.50 g of aluminum to  $\text{KAl(OH)}_4$ . Write the balanced equation first.

\_\_\_\_\_ moles

\_\_\_\_\_ g

2. Calculate the number of milliliters of 9.0M  $\text{H}_2\text{SO}_4$  that you would need to convert 0.070 moles of  $\text{KAl(OH)}_4$  to  $\text{K}_2\text{SO}_4$  and  $\text{Al}_2(\text{SO}_4)_3$  according to the following equation:



3. How many moles and how many grams of potassium alum,  $\text{KAl(SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ , can be prepared from 5.00 g of aluminum?

4. If 75.0 g of potassium alum is obtained from 5.00 g of aluminum what is the percent yield?

5. How many grams of aluminum were used to prepare 20.0 g of potassium alum if the percent yield was 81.5%?

Name \_\_\_\_\_ Date \_\_\_\_\_ Score \_\_\_\_\_

### Data Sheet

*WHETHER RECORDING DATA OR SHOWING CALCULATIONS, REMEMBER TO INCLUDE MEASUREMENT UNITS WITH EACH NUMERICAL VALUE.*

1. Mass of cleaned Al strip \_\_\_\_\_

2. Moles of Al \_\_\_\_\_

3. Theoretical moles of alum \_\_\_\_\_

4. Theoretical yield of alum \_\_\_\_\_

5. Mass of alum obtained \_\_\_\_\_

6. Percent yield of alum \_\_\_\_\_

Instructor's Approval of Crystals \_\_\_\_\_

### Calculations:

Show your method in detail. The number of significant figures in your answers must be correct.

Name \_\_\_\_\_ Date \_\_\_\_\_ Score \_\_\_\_\_

**Postlaboratory Assignment**

*FOR FULL CREDIT, SHOW DETAILED CALCULATION SETUPS. REMEMBER TO FOLLOW THE SIGNIFICANT FIGURES CONVENTION, AND TO SHOW MEASUREMENT UNITS FOR EACH QUANTITY.*

1. What causes the Al clippings to rise and fall as they are being dissolved in the KOH solution?
2. The synthesis of alum proceeds in several reaction steps. The mole ratios of reactants and products can be found by combining the written equations for these separate reactions into an overall equation. Balance the overall equation for the synthesis of alum,  $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ , from aluminum, potassium hydroxide, sulfuric acid and water. **HINT:** Add together the four equations given in the introduction, canceling out any molecules or formula units that appear on both sides.



3. Look at the equations given in the experiment; identify the oxidation-reduction reactions in which the oxidation number of an element changes.
4. Explain why aluminum reacts slowly with dilute acids.
5. Balance the following equation:







