

## Synthesis of Alum: $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$

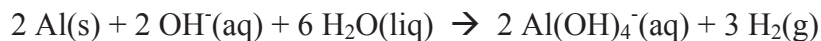
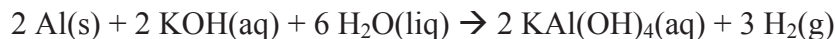
**Hazard Warning: The sodium hydroxide used in this experiment is highly corrosive. If you get it on your skin, wash immediately. If your skin feels slippery, that is a sign that you have gotten the sodium hydroxide on you.**

### Introduction

The purpose of this experiment is to use aluminum from an aluminum can to synthesize a chemical compound, alum, which is hydrated potassium aluminum sulfate,  $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ .

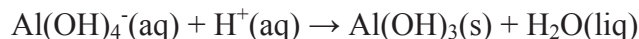
Aluminum beverage cans generally have a thin coating of plastic on the inside that protects the aluminum from the corrosive action of the chemicals in the beverage. The outside usually has a thin coating of paint. These coatings must be removed before any chemical reactions with the metal can be carried out. The coatings may be effectively scraped off with a metal pan cleaner. A cleaned piece of metal is then dissolved in a potassium hydroxide solution according to the following complete, balanced equation:

The full and net ionic equations are:



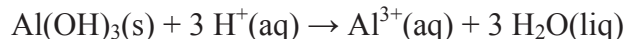
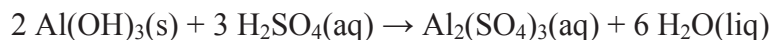
The  $\text{Al}(\text{OH})_4^-$  ion is a complex ion called "aluminate." After filtration to remove residual plastic and paint decomposition products, the alkaline solution of  $\text{Al}(\text{OH})_4^-$  is **clear** and **colorless**. The  $\text{H}_2$  is evolved as a gas and mixes with the atmosphere. The chemical species in solution are potassium ions ( $\text{K}^+$ ) and aluminate ions [ $\text{Al}(\text{OH})_4^-$ ] ions (plus any unreacted  $\text{KOH}$ ).

Sulfuric acid is now added and two sequential reactions occur. Initially, before the addition of all the acid, insoluble aluminum hydroxide is formed,



$\text{Al}(\text{OH})_3$  to give a thick, white, gelatinous precipitate of aluminum hydroxide. As more sulfuric acid is added, the precipitate of  $\text{Al}(\text{OH})_3$  dissolves to form soluble  $\text{Al}^{3+}$  ions.

The full and net ionic equations are:



to give aluminum ions,  $\text{Al}^{3+}$ , in solution. The solution at this point contains  $\text{Al}^{3+}$  ions,  $\text{K}^+$  ions (from potassium hydroxide), and  $\text{SO}_4^{2-}$  ions (from sulfuric acid). On cooling, crystals of **hydrated** potassium aluminum sulfate,  $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$  (or alum) are very slowly deposited. In the experiment the crystallization process is speeded up by providing a small "seed crystal" of alum for the newly forming crystals to grow on. Cooling is needed because alum crystals are soluble in water at room temperature.

The full and net ionic equations are:



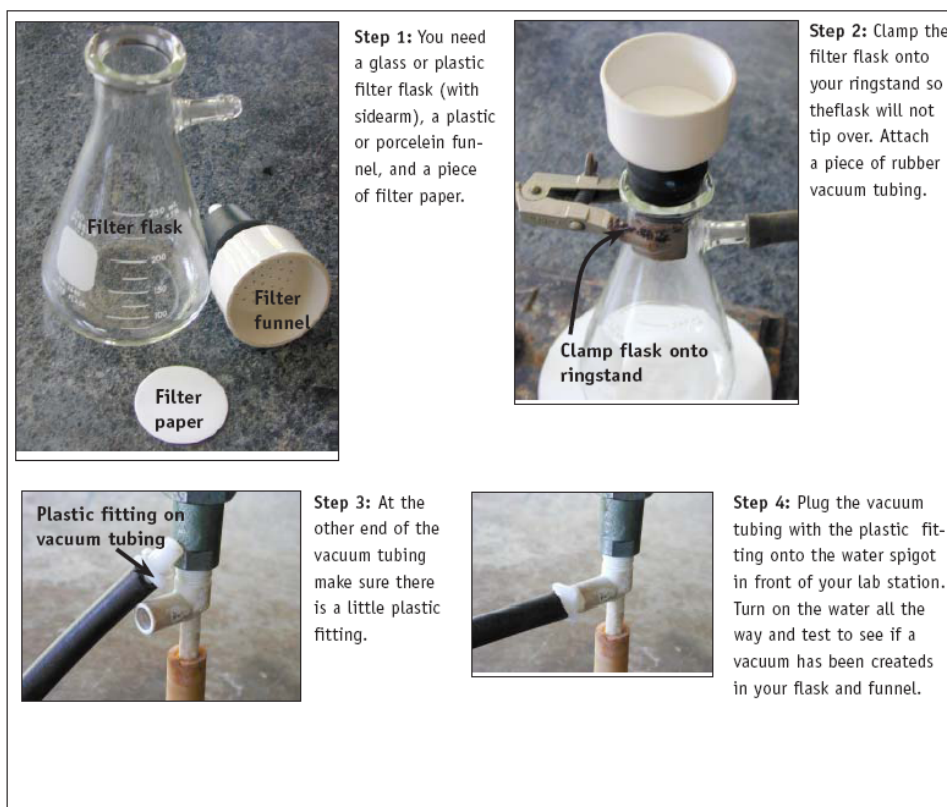
Finally, the crystals of alum are removed from the solution by vacuum filtration and washed with an alcohol/water mixture. This wash liquid removes any contamination from the crystals but does not dissolve them. It also helps to dry the crystals quickly, because alcohol is more volatile than water.

## EXPERIMENTAL PROCEDURE

*Your goal is to make this compound and to determine the percent yield. Before starting, you must tell your instructor at which steps below you will record accurate weights.*

1. Clean all glassware.
2. Pierce the can at the lower end of the side with the point of a pair of scissors. Cut around so that the sides of the can are cut out. Deposit the waste aluminum scraps left over in the box provided.
3. Lay the rectangular piece of aluminum (the sides of the can) on the bench and scour both sides with the pan scrubber provided. Make sure that an area of 2" x 2(1/4)" is clean on both sides.
4. Wipe the metal clean with a paper towel and cut out a clean piece that is about 2" x 2 1/4".
5. Take this piece to the analytical balance and weigh it. If the piece weighs more than 1 g, cut small pieces off until it weighs about 1 gram.
6. Cut the weighed piece into smaller pieces, and place them in a clean 250 mL beaker. Do not lose any metal bits.
7. Using a graduated cylinder, add 50 mL of 1.4 M KOH. **DO IT IN THE HOOD!**
8. Set up a ring stand, ring clamp, gauze, and bunsen burner. Place the beaker on the gauze and heat the beaker on a low flame. It is not necessary to boil it. The aluminum will take about 20 minutes to dissolve.
9. While it is dissolving, set up an apparatus for *gravity filtration* (as described by the figure on page A-3). Place a clean funnel, with a piece of folded filter paper, on a 125 mL Erlenmeyer flask.
10. When the aluminum has dissolved (as evidenced by the lack of bubbles of H<sub>2</sub> gas given off), gravity filter the solution. Only fill the funnel to within 1/2" of the top of the paper. Use a glass rod to "guide" the solution into the paper (as demonstrated by your instructor). The solution in the Erlenmeyer flask should be both clear and colorless at this point.
11. Allow the flask to cool. While it is cooling, wash the funnel and beaker with lots of tap water to remove any potassium hydroxide.
12. When the solution is reasonably cool, add 20 mL of 9 M H<sub>2</sub>SO<sub>4</sub> (with a graduated cylinder) quickly and with care. It is important that you swirl the flask as you add the acid. The solution will get quite warm. If there are any white flecks left in the solution after the addition of the H<sub>2</sub>SO<sub>4</sub>, place the flask on the Bunsen burner apparatus and warm it with swirling until all of the solid material has dissolved.
13. Make an ice bath by putting ice and water into a 600 mL beaker.

14. Allow the flask to cool a little and then place it in the ice bath and allow it to cool for an additional 5 minutes.
15. If alum crystals have not started to form, scratch the inside walls of the flask with a stirring rod. This provides sites at which crystallization can begin, followed by crystal formation throughout the liquid. Swirl the flask when you notice the onset of crystal formation and allow it to cool in the ice bath for another 10 minutes.
16. While the solution is cooling, pour 50 mL of 50% alcohol/water mixture into a test tube and place it in the ice bath to cool.
17. Set up the *vacuum filtration* apparatus (Buchner funnel, see figure below) as illustrated by your instructor. Pour some distilled water onto the filter.
18. Remove the flask containing the alum crystals from the ice bath, swirl so that all the crystals are dislodged, and pour quickly into the Buchner funnel. Keep swirling and pouring until all the solution and crystals are transferred to the funnel. The water aspirator should be kept going all through this process.
19. Pour about 10 mL of the cooled alcohol/water mixture into the flask. Swirl the flask and pour mixture into the funnel to transfer any remaining crystals.
20. Store your crystals in a beaker until next week to ensure that they dry completely.



**Figure** This figure illustrates the way to set up and carry out a vacuum filtration.

## REPORT

Prepare a brief report describing your observations during the experiment. Report the actual yield, the theoretical yield and the percent yield.

