

# Synthesis of Alum from Aluminum

*Taken from Central Oregon College Chemistry Manual*

**OBJECTIVES:** To carry out a series of reactions to transform a piece of aluminum foil into crystals of alum and to gain familiarity with using stoichiometry to determine the yield for the reaction.

**SAFETY AND DISPOSAL:** Strong acids and bases are corrosive and should be handled with care. Immediately wipe up any spills and wash hands with plenty of soap and water. Work in the mini-hoods whenever possible to avoid breathing in fumes. All solutions can be disposed of down the sink with running water. The alum can be safely disposed of in the trash but has a number of uses and can be used for other laboratories, place alum crystals into the designated container.

**INTRODUCTION:** Aluminum is a material that you may have had some experience with and paid little attention to, but has some very interesting characteristics. Here are some fun facts about aluminum metal:

1. Aluminum is the third most abundant element in the earth's crust.
2. The supply of aluminum ore is not inexhaustible.
3. The winning, or extraction of the metallic form from an impure ionic source, of aluminum from aluminum ore is very costly from an energy point of view.
4. The above point explains why Napoleon III used aluminum dinnerware for his state dinners. Lesser guests were served on plates of gold or silver.
5. The process of obtaining pure metallic aluminum from aluminum oxide in a reasonably energy efficient manner, which made it possible for aluminum to be the inexpensive metal we know today, was developed in a home laboratory shortly after the chemist finished his undergraduate degree.
6. Aluminum is an exceedingly useful material, in part because under everyday environments it is quite indestructible. Thus, an aluminum beverage can lying alongside a highway has an estimated lifetime of 100 years.
7. From a chemist's point of view, aluminum is very reactive.

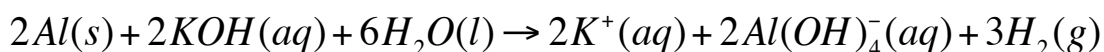
Facts 6 and 7 seem to contradict each other. How can a very reactive substance be indestructible under many circumstances? The answer to this conundrum is due to the fact that aluminum reacts with oxygen in earth's atmosphere to produce a very thin yet unreactive coating of aluminum oxide ( $\text{Al}_2\text{O}_3$ ) on the surface of the aluminum. This "adherent oxide coating" is very thin, transparent, and it protects the bulk of the aluminum from further oxidation. Since you can see through it to the shiny metal surface underneath, it looks like there is no coating on the metal at all. This oxide coating does react with water slowly, so aluminum used for beverage

cans must be coated with a polymer to keep them from dissolving.

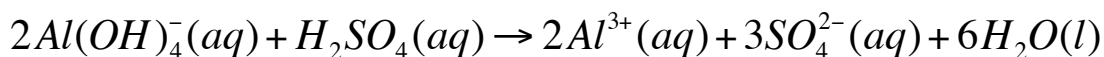
Facts 2, 3, 6, and 7 suggest that it is both important and possible to recycle aluminum. One way scrap aluminum could be recycled is to transform it into a useful chemical. One such chemical is potassium aluminum sulfate dodecahydrate,  $KAl(SO_4)_2 \cdot 12H_2O$ , commonly called alum. In practice, this would be an expensive method for synthesizing alum; aluminum is most often recycled for reuse as metallic aluminum.

The reactions involved in converting aluminum into alum are as follows:

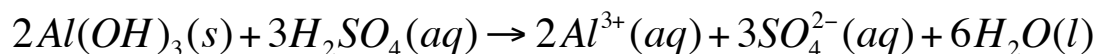
1. Reaction of aluminum with KOH



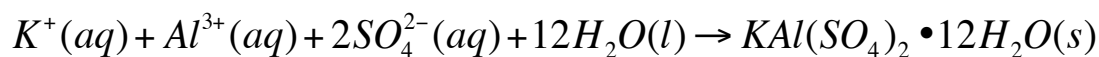
2. Initial addition of  $H_2SO_4$



3. Further reaction with  $H_2SO_4$



4. Precipitation of alum on cooling



Although the product we seek today is listed in equation 4 as being a solid, in reality it is slightly soluble in water, and will gradually crystallize from water when the temperature of that aqueous solution is lowered. This solid can be separated from the solution by filtration.

If you assume that all of the aluminum that was dissolved in the first reaction is converted to alum then the mass of aluminum that was used in the first step can be used to predict how much alum you should theoretically be able to recover in this final step. Aluminum is the limiting reagent in today's lab, with all other reagents being used in excess. The amount you actually recover may be different from the theoretical amount for a variety of reasons, including factors outside your control such as side reactions and reactions that do not go to complete as well as due to experimental errors. The ratio of what you actually recovered over the theoretical yield is one measure of reaction efficiency. This difference is called the percent yield and is defined as follows:

$$\% \text{yield} = \frac{\text{actual yield (g)}}{\text{theoretical yield (g)}} * 100$$

#### EXPERIMENTAL PROCEDURE:

Obtain approximately 1 g of aluminium foil, making note of the precise amount. Tear the foil into small pieces and place in a beaker. Add 50 mL of 1.4 M KOH to the foil. Bubbles of hydrogen gas should start to be visible, a product of the reaction between Al and OH<sup>-</sup>.

Heat the beaker gently until there is no more sign of reaction occurring. This should take no more than 30 minutes. Continue heating until the final volume of the solution is about 30 mL. If the reaction volume drops below this level, add water to reach the desired final volume.

Assemble a vacuum filtration apparatus as shown in the demonstration setup in the back of the lab. Clamp the apparatus to a ring stand so that it does not tip over. If it falls, you will both lose product and risk breaking the funnel. Place a piece of filter paper in the funnel such that it completely covers all of the holes in the funnel and lies flat. Connect the side arm of the funnel to the aspirator, lightly wet the filter paper to seat it and turn on the aspirator. Pour all of the hot solution from your beaker through the funnel. Rinse the beaker twice with 5 mL portions of distilled water, collecting each rinse. Add no more water than is absolutely necessary to rinse the apparatus.

Pour the filtrate into a clean 250 mL beaker. Rinse the filter flask with 10 mL of distilled water and add to the filtrate. Allow the solution to stand until it reaches room temperature.

While stirring, carefully add 20 mL of 9M H<sub>2</sub>SO<sub>4</sub> to the cooled solution. White Al(OH)<sub>3</sub> should first appear in the beaker, which in turn will redissolve as additional acid is added. If necessary, warm the solution gently to completely redissolve the Al(OH)<sub>3</sub>.

Place the beaker in an ice bath. Allow the solution to chill for 15 minutes, during which time alum crystals should form. Be patient. The more slowly crystals form, the larger they will be. Filter the alum crystals using the same filtration system as you used before, using a fresh piece of filter paper. Recover as much of the alum onto the filter paper as possible. A rubber policeman can help with the transfer process. Use two 10 mL of a 50-50 solution of pre-cooled ethanol-water to rinse any remaining crystals from the beaker. Allow the crystals to dry by continuing to pull air through the filter apparatus for an additional ten minutes.