

Experiment 4: Synthesis of Alum from Scrap Aluminum

Objective: In this experiment, you will be converting the aluminum metal from a beverage can into the chemical compound potassium aluminum sulfate, $KAl(SO_4)_2 \cdot 12 H_2O$, commonly referred to as *alum*.

Introduction

It had been a good year for the O'Keefe farm with just the right amount of sun and rain. In fact, the cucumbers had grown so well that Richard was afraid he wouldn't be able to sell them fast enough. Faced with the prospect of a warehouse full of rotten cukes, he contemplated the idea of preserving them and selling them as "O'Keefe's Farm Fresh Pickles". He discussed the plan with his wife, Diane, who agreed it was a good idea and said she had a delicious pickling recipe that had been handed down to her from her mother.

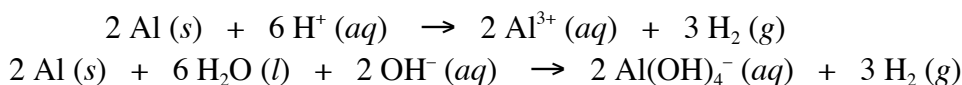
"I recall one of the ingredients was alum. I think it helps to keep the pickles firm and crisp," Diane said.

"It just so happens that I know of a recipe for making alum from aluminum cans," Richard said. "We can use some of those soda pop cans that have been piling up, and that should save us some money."

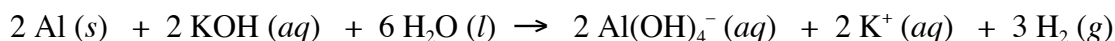
"How can you convert aluminum metal into an aluminum salt?" Diane asked.

Richard explained the chemistry behind the process:

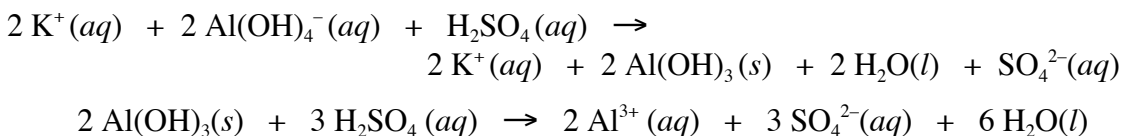
One of the interesting properties of aluminum is that it is **amphoteric**, meaning it will dissolve in both strong, aqueous acids and strong, aqueous bases (see Tro, pp 758-759). In both cases, the formation of hydrogen gas is observed:



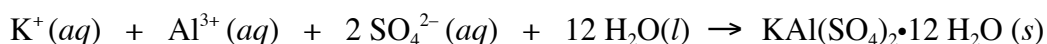
The first reaction that needs to be carried out is that of aluminum and potassium hydroxide, KOH:



Adding sulfuric acid, H_2SO_4 , to the resulting solution will cause the compound $Al(OH)_3$ to precipitate; however, $Al(OH)_3$ is also amphoteric and will re-dissolve when more acid is added:



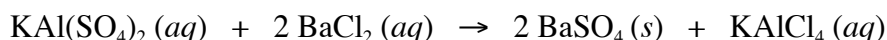
Crystals of the double salt $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O} (s)$, or alum, will form upon cooling this final solution since the solubility of alum in water decreases as the temperature is lowered:



“But how will we know that everything really worked ok and that it’s really alum that you produced? I think we need to implement some quality control measures,” Diane observed. “In order to confirm that your synthesis of alum resulted in the desired product, we need to perform a **qualitative analysis** of the compound.”

Qualitative analysis is the name given to the process whereby the identities of the elements or substances present in a sample are determined through the use of simple chemical tests (see Tro, pp 752-754). In this case, chemical reactions will be performed with an alum sample that will confirm the presence of K^+ , Al^{3+} and SO_4^{2-} .

In the first test, alum will be reacted with barium chloride in an aqueous solution, which will result in the following chemical reaction:



The precipitation of solid material, BaSO_4 , from a solution of alum upon reaction with barium chloride is a positive test for the presence of sulfate ion, SO_4^{2-} .

The second test is performed to confirm the presence of potassium and is a flame test. Potassium is volatilized at the very high temperature of a flame (about 1000°C) at which point it imparts a bluish-purple color to the flame. After a few seconds in the flame, the sulfur dioxide will be driven off of your alum sample, and the solid material remaining will consist of aluminum oxides.

The third test confirms the presence of aluminum ion and involves its reaction with potassium hydroxide. A wispy, gelatinous precipitate of $\text{Al}(\text{OH})_3$ will form upon addition of a small amount of KOH to the aqueous alum solution. Further addition of KOH will cause the precipitate to re-dissolve.

“We should also make sure that we’re really saving some money. We’ll need to calculate the percent yield of the process once we’ve made the alum,” Diane said.

When carrying out chemical reactions, it is of interest to know what percentage of the starting material is recovered in the form of the desired product. It is possible to calculate this **percent yield** in the following manner (see Tro, pp 133-139):

1. Calculate the number of moles of reactant, in this case, aluminum.

$$\text{number of moles Al} = \frac{\text{mass of Al (g)}}{\text{molar mass of Al (g/mol)}}$$

2. From the stoichiometry of the reaction, determine the expected number of moles of product. A review of the above reactions reveals that there is a 1:1 relationship between the aluminum containing reactant and the aluminum containing product in all cases. Therefore, one mole of aluminum metal should produce one mole of alum.

$$\text{number of moles alum} = \text{number of moles Al} \cdot \frac{(1 \text{ mole alum})}{(1 \text{ mole Al})}$$

3. From the expected number of moles of product, calculate the expected mass of product or the **theoretical yield**.

$$\text{theoretical yield} = (\text{number of moles alum})(\text{molar mass alum, g/mol})$$

4. Calculate the **percent yield** by dividing your actual yield by the theoretical yield and multiplying by 100.

$$\text{percent yield} = \frac{\text{mass of alum (g)}}{\text{theoretical yield (g)}} \times 100$$

O'Keefe's Farm Fresh Pickles

Take 2 gallons washed/chunked pickling cucumbers. Place in a crock or other large non-aluminum container. Measure enough water to cover the cucumbers, then add 1 pint of pickling salt to water, then bring the water to a boil. Finally, pour boiling brine (salt water) over the cucumbers. Then weigh cucumbers down with a plate so that they stay submerged under the brine. Allow this to stand for 1 week.

After 1 week, drain the pickles and discard the brine. Cover pickles with fresh boiling water and let stand for 24 hours.

Drain, but save the liquid. Add a walnut sized piece of **alum (6 grams) to the liquid**. Bring to a boil and then pour over the pickles. Let stand for 24 hours. Note: you can omit the alum if you wish, but alum does help to keep the pickles crisp.

After 24 hours, drain the liquid and discard it. Boil together 6 pints of vinegar, 2 pints water, 2oz of mustard seed, 4 cups of sugar, 5 oz of cinnamon sticks, 1 oz whole allspice. Add to pickles through a strainer. Let stand 24 hours.

Bring to boiling point, but do not boil. Wash jars clean and keep hot until needed. Prepare lids as manufacturer directs. Pack pickles into hot clean jar leaving 1/2 inch of headspace. Wipe rim with clean damp cloth and seal. Continue until all jars are sealed. Process in a boiling bath for 10 min.

Taken from:

<http://www.mapickle.com/recipe.htm>

(website no longer in service)

Procedure

Synthesis of alum

Wear safety glasses at all times!

CAUTION: KOH is highly caustic; take extreme care not to splatter and avoid contact with skin and eyes.

Obtain a piece of scrap aluminum from a beverage can and cut it into thin strips. Accurately weigh out about 0.5 g of these strips and place them in a 150 mL beaker. Carefully add 25 mL of 1.5 M potassium hydroxide, KOH, solution. If any of this solution should spill, clean it up immediately.

Proceed with the reaction **in a fume hood**.

Set the beaker on a hot plate and turn the dial to medium high. Heat the mixture for about 30 minutes in order to dissolve as much of the aluminum as possible. Stir frequently in order to keep the metal pieces from floating to the top of the froth. If necessary, add water to keep the volume constant. Do not allow bubbling to become so vigorous that the froth spills over the side of the beaker. The hydrogen gas (**flammable!**) generated by the reaction will be carried away by the suction of the fume hood. The reaction may splatter so heating should be slow and care should be exercised at all times.

After most of the metal has dissolved, the fizzing around each piece should stop. There may still be some undissolved solids present such as paint and varnish particles. Turn the hot plate off and gravity filter the hot solution through filter paper in a funnel (see TECH Section I.G.). Rest the funnel in a wooden funnel holder. Collect the filtrate in a 250 mL beaker and cool to room temperature.

Acidify the solution slowly by carefully adding 10 mL of 9 M H₂SO₄ (**caution! wear gloves**) in 2-3 mL portions with continuous stirring. **Caution! Considerable heat is generated by this reaction.** A precipitate of aluminum hydroxide, Al(OH)₃, will form during the initial addition of acid; however, this solid will re-dissolve upon further acidification.

If any white lumps still remain after addition of all the sulfuric acid, gently heat the mixture, with stirring, until the solution becomes clear. Remove the heat and let the solution cool for a few minutes. Make an ice bath by filling a large beaker ¹/₂ - ³/₄ full with ice and adding cold water. Place the cooled beaker in the bath for about 20 minutes. Crystals of alum should form in the beaker. If no crystals grow, stir the solution and try to induce crystal formation by scratching the inside of the beaker with a glass rod.

Set-up a filter flask as shown in TECH Section I.F. Turn on the vacuum and wet the filter paper with a small amount of water, pushing down on the funnel to obtain a better vacuum seal. Pour your suspension of crystals onto the filter paper. Allow the liquid to drain completely, then wash out the beaker with 10 mL of 50/50 ethanol/water solution in order to completely transfer any crystals still left in it. Pour this mixture (in which the alum is not very soluble) over the crystals in the funnel and again let the liquid drain. Finally, wash the crystals with 10 mL of ethanol. Leave the vacuum on for about 5 minutes in order to draw air through the crystals to dry them. Carefully scrape the crystals off the filter paper and onto a watch glass. Pour the liquid filtrate into the appropriate **Laboratory Byproducts** jar.

Label a clean, *dry* vial with the word "Alum", your name and the date, and leave a space to enter the total mass produced. Weigh the vial and cap to the nearest 0.001 g and record the weight in your notebook. This is the **tare weight** of the container. Transfer your dried alum crystals to the vial, weigh again to the nearest 0.001 g and record this weight in your notebook. Enter the total mass of alum produced on the label of the vial.

Chemical tests for qualitative analysis of alum

Transfer enough crystals of alum to just cover the pointed end of your spatula to a small test tube, and dissolve them in about 6 drops of distilled water (use a medicine dropper). Be sure all the alum is completely dissolved. Remove a drop of this solution from the test tube using a glass stirring rod, and transfer it to a piece of blue litmus paper. Record the color of the litmus paper. Add one or two drops of 0.5 M BaCl₂ solution to the alum solution in the test tube, and record your observations. Pour the contents of this test tube into the **Barium Laboratory Byproducts Jar**.

Caution! Ethanol is flammable. Before lighting a Bunsen burner, check to be sure that there is no ethanol in the vicinity.

Place a small amount of your *solid* alum product on a watchglass and carry it to the section of the laboratory which has been set up for the flame tests. Your instructor will provide you with a wire loop. Clean the loop by dipping it in a solution of concentrated HCl and placing it in the Bunsen burner flame until it glows red hot. **Caution: Concentrated HCl is extremely corrosive and should be handled with care. If any HCl spills on your skin, wash the area immediately with running water.** Now, touch the loop, while still hot, to the sample of alum on the watchglass such that some of the crystals stick to the loop. Hold the crystals in the flame for several seconds (until the solid glows), and record your observations (especially the color of the flame).

Obtain approximately 1 mL of 1.4 M KOH solution. Place 1 drop of this solution in a small test tube and add 9 drops of water. This dilution will result in a solution which has a concentration of 0.14 M KOH. Transfer a spatula tip full of alum crystals to a small, clean test tube, and completely dissolve the alum in about 5 drops of water. While stirring with a small glass rod, add 1 to 4 drops of 0.14 M KOH solution and observe what happens. Now add 1 to 4 drops of the more concentrated 1.4 M KOH solution, and observe what happens.

Clean up your work bench, fume hood and sink before leaving. Do **not** leave scraps of aluminum can in the sink.

Hand in the labeled vial that contains your product to your instructor.

Calculations

1. Following the procedure given in the introduction, calculate the **theoretical yield** of alum and the **percent yield** that you obtained from the preparation.
2. Calculate the mass of alum recovered per gram of aluminum by dividing the mass of alum crystals by the initial mass of aluminum metal.

Questions

1. Discuss your observations from the first chemical test. What did the test with litmus paper tell you? What conclusion can you draw from the test with BaCl_2 ?
2. What did you see in the flame test, and does this confirm the presence of potassium ion in your sample? What do you think would happen if you could dissolve the solid that remains *after* the flame test and added BaCl_2 solution to it? Explain.
3. What did you see in the reaction of alum with KOH , and did it confirm the presence of aluminum? What did you see when more KOH was added? Write a chemical equation for the reaction of the aluminum hydroxide compound, $\text{Al}(\text{OH})_3$, with KOH . [**Hint:** see the equations in the introduction to the experiment for ideas on possible products for this reaction.]
4. The O'Keefe's have 50 gallons of cucumbers for pickling. The 1.5 M KOH solution costs \$5 per liter, 9 M H_2SO_4 solution costs \$7.50 per liter, and ethanol costs \$25 per liter. A 50 g jar of alum from the grocery store costs \$7.89. Do you think it's worth the time and trouble for Richard and Diane to make their own alum from discarded aluminum cans? Explain your answer showing all calculations.
5. a) List sources of error that would contribute toward lowering the percent yield of alum.
b) List sources of error that would contribute toward producing a percent yield of greater than 100%.