Synthesis of Alum


Introduction

Aluminum cans are often recycled to make more aluminum products. In this experiment, you will synthesize a compound called *alum*, starting from the aluminum in soda cans. The name *alum* refers to a class of chemical compounds that are valuable for water purification, making explosives, tanning processes, deodorant sticks, medical astringents, and as shown here, crisping pickles.

What you have learned so far about analogical reasoning and how to construct a strong scientific argument based on evidence, reasoning and claims are directly transferrable to this lab. Thus, as you synthesize alum, try to use analogical reasoning and modeling to help you think about what is happening at the atomic scale. In addition, as you make chemical observations in lab, think about the evidence you are gathering that will help you make claims and construct a scientific argument. Your lab report will include the overall yield of the reaction, a discussion about the observations made in lab, and a hypothesis about how the reaction conditions in each step of the synthesis of alum changes the chemical state of the aluminum atoms.

Your lab report will be due the day following your next lab session. Refer to the end of the procedure (11. Recrystallization) for the reasons behind this.

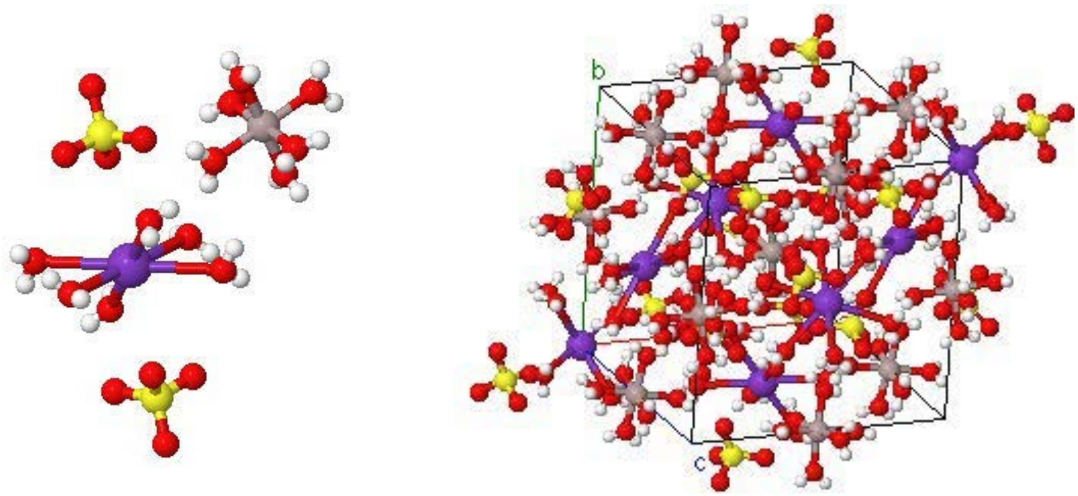
Goals:

1. To synthesize alum from aluminum soda cans.
2. To make observations about each intermediate chemical reaction in the synthesis and to describe what is happening on the atomic scale.
3. To calculate the theoretical yield and percent yield of the synthesis reaction.
4. To construct a discussion about how the interactions of aluminum with other atoms and molecules changes throughout the synthesis of alum.
5. To think about the chemical transformations that occur at the atomic scale.

In this experiment, you will produce alum from an aluminum can in a multi-step synthesis reaction summarized by the chemical equation below:

$$2Al(s) + 2KOH(aq) + 4H_2SO_4(aq) + 22H_2O(l) \longrightarrow \underbrace{2[KAl(SO_4)_2\bullet 12H_2O](s)}_{\textbf{alum}} + 3H_2(g)$$

The name "alum" actually describes several related compounds. The compound that you will synthesize today is potassium aluminum sulfate dodecahydrate [$KAl(SO_4)_2\bullet 12\ H_2O$]. Alum is a type of compound called a *double salt* because there are two different cations (K^+ and Al^{3+}) that crystallize together in a single solid. The dot and the 12 H_2O molecules in the formula signal that the compound is "hydrated". These water molecules are included in the unit formula and contribute to the mass of a single molecule of the compound. Therefore, the molecular weight of potassium aluminum sulfate dodecahydrate is 474.37 grams/mole.



A. B.

Figure 1. Crystal structure of [$KAl(SO_4)_2$•$12H_2O$]. (K = purple, Al = gray, S = yellow, O = red, H = white). A. $KAl(SO_4)_2 12$•H_2O. B. The packing of [$KAl(SO_4)_2$•$12H_2O$] in a crystal. [Source: Cambridge Structural Database (file: EVUWAV) accessed 10/2016.

Percent yield and theoretical yield:

Knowing the molar ratios provided by a balanced chemical equation, you can predict the amount of product (in this case, the amount of alum [$KAl(SO_4)_2$•$12H_2O$] you will produce based on the amount of each reactant used. This is called the **theoretical yield**. In this synthesis, the reactants, potassium hydroxide and sulfuric acid will be used in excess of the molar ratio needed to complete the reaction.

That means that the *limiting reactant will be the amount of aluminum metal* that you start with.

In a synthesis reaction it is difficult to obtain the mass of product predicted by a calculation of the theoretical yield. There are a number of possible obstacles. Because this is a multi-step reaction, several intermediate products are formed along the way, each containing aluminum. Different steps require transfers of the reaction between different glassware, filtration, stirring, etc., all of which can lead to the loss of some of the intermediate compounds necessary for forming the final alum product. Therefore, it is useful to calculate the percent yield which provides a comparison of the actual yield and the theoretical yield:

$$\text{Percent yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

Pre-lab Assignment

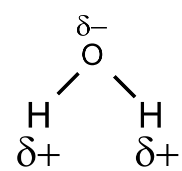
In your lab notebook, write answers for questions 1-5 below. You must complete the pre-lab before coming to the lab meeting or you will not be permitted to go into the laboratory.

1. Write a two to three sentence introduction to the lab.

2. Figure 1 on page 2 shows a model (i.e. a type of analogy) of the arrangement of atoms in the solid state structure of alum [$KAl(SO_4)_2\bullet 12H_2O$]. For example, you can see that the purple spheres representing potassium ions (K^+) are surrounded by 6 water molecules. Since alum is a salt (also called an electrolyte) it is soluble in water. When the solid dissolves in water, the cations and anions are "solvated" by water. Solvation helps to stabilize the ions in solution and prevents the cations and anions from recombining. Although water is neutral, there is a slight negative charge on the oxygen and a slight positive charge on each

hydrogen (see picture here).

Draw a picture to represent the solvation of cations (Al^{3+} and K^+) and anions (SO_4^{2-}) when alum dissolves in water.



3. Create a safety information table that includes the chemicals used in the lab, the hazards associated with them, and any safety handling precautions.

4. What volume of 1.5M potassium hydroxide (KOH) solution is needed to react with 1.0 g of aluminum (Al) according to the balanced chemical equation on page 1? (See the sample "worked problem" below.)

5. What volume of potassium hydroxide should be used to react with 1.0 g of aluminum if you want a two-fold molar excess of potassium hydroxide?

Worked Problem: Starting with approximately 1 g of iron, how much of a 1.55M solution of H_2SO_4 would be needed to react according to the equation?

$$Fe(s) + H_2SO_4(aq) \rightarrow FeSO_4(aq) + H_2(g)$$

Step 1. The piece of iron weighed exactly 1.060g. Therefore:

(1.060g Fe) (1.00 mole Fe / 55.845g Fe) = 0.0190 moles Fe

Step 2. According to the balanced equation for the reaction, for every 1 mole of Fe, we need 1 mole of H_2SO_4. Therefore:

we would need 0.0190 moles of H_2SO_4.

Step 3. The volume of 1.55M H_2SO_4 solution required can be calculated:

(0.0190 moles H2SO4) (L/1.55 moles) (1000 mL/ L) = 12.2 mL

Scientific Question: How do changes in chemical state (solid to solute to crystal) affect the way aluminum interacts with other chemicals?

- It can be helpful to include the observations about the reactants, products and relative rates of the different steps in the alum synthesis and drawings of aluminum at the atomic scale.

There are a variety of ways to answer this question. In all cases, your argument needs to be sufficiently supported with evidence you gather throughout the experiment. Be sure to carefully observe and record all steps of the synthesis and pay special attention to the questions as you work through the procedure.

Laboratory Guide

Please pair up with another student when doing lab work. If there is an odd number of participants in lab, one group may be permitted to have three people. Record all your observations in your own lab notebook.

Your lab work will also involve working in partnership on certain activities, answering questions, discussing observations, or analyzing results. As you go through the experimental guide, you will notice there are questions that are set off in the guide (i.e. "**Q:**"). For example:

Q: Companies apply a polymer coating to the inside of the aluminum soda can. Can you guess why?

You are required to respond to these questions in your lab notebook. Our expectation is that you write enough to give an indication of what you were thinking about. You do not have to write down the question AND answer, but you must address the answer, for example: "polymer coating on the inside is probably for ….."

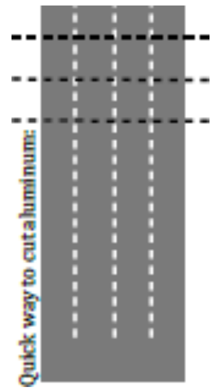
Goggles are required at all times in the lab. There are no exceptions. Gloves and footwear are available. If you have questions about safety, please do not hesitate to ask your laboratory instructor. This lab has portions of the procedure that must be completed under the hood.

Chemicals:	**Materials:**
aluminum from soda can	**Supplies and Equipment*:**
1.5 M potassium hydroxide (KOH)	electronic balance
9.0 M sulfuric acid (H_2SO_4)	steel wool
ice	glass gravity filtration funnel
50%:50% ethanol:water mixture	gravity filtration filter paper (11 or 12.5 cm qualitative)
	Bunsen burner and ring stand
	Buchner funnel and vacuum flask
	Buchner funnel filter paper (9.0 cm)
	Various glassware, including large beaker for ice bath

*(*illustrations of how to set up filtration equipment are provided in procedure)*

Laboratory Procedure

1. Obtain a strip of aluminum from a soda can. Using steel wool, scrub both sides of the strip to remove the paint from the outside and the polymer coating from the inside.



Q: Companies apply a polymer coating to the inside of the aluminum soda can. Can you guess why?

2. Use scissors to cut the strip into tiny pieces and weigh out about 0.7 g of the metal. It is not important to have exactly 0.700 g of aluminum, but it is important to record the **exact** mass to ±0.001 g in your lab notebook.

3. Using your actual mass of aluminum and the concentration of KOH, calculate the volume of KOH needed to add a two-fold molar excess to the aluminum. (You should not have to add more than 65 mL of KOH. If your calculation result is >65 mL, see your lab instructor.)

a

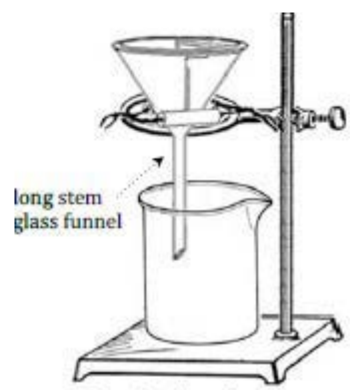
$$2Al(s) + 2KOH(aq) + 6H_2O(l) + heat \longrightarrow 2KAl(OH)_4(aq) + 3H_2(g)$$

4. Place the aluminum pieces in a 250 mL beaker and SLOWLY add the two-fold molar excess of KOH solution. DO NOT use an Erlenmeyer flask.

Q: What evidence do you have that a gas is being produced during the reaction?

5. When the initial bubbling and foaming has slowed, heat the solution over a Bunsen burner <u>very gently</u>. DO NOT BOIL. If the level of the liquid drops to less than one quarter of the original level, add some distilled water. After the fizzing has completely ceased (10-15 minutes) and you no longer see chunks of aluminum in the solution, remove the beaker from the flame.

Q: Why can you no longer see the aluminum once the reaction is complete?



Gravity filtration

6. While the solution is still hot, filter it by gravity filtration (see picture at right for setup), to remove black insoluble impurities. Discard filter paper and save the filtrate collected in the beaker.

b

$$2KAl(OH)_4(aq) + H_2SO_4(aq) \longrightarrow 2Al(OH)_3(s) + K_2SO_4(aq) + 2H_2O(l)$$

7. Cool the solution to room temperature and then slowly add 15 mL of 9 M H_2SO_4. Stir the mixture continuously. Record your observations.

Q: After the reaction occurs, what is the chemical formula of the solid you are observing? What chemical species are present in the solution?

c $2Al(OH)_3(s) + 3H_2SO_4(aq)$ + heat $\longrightarrow$ $Al_2(SO_4)_3(aq) + 6\,H_2O(l)$

8. Using a Bunsen burner, heat the mixture gently for 10 minutes. Watch carefully for splattering. The solution should become clear. If any solid is seen, filter the warm mixture again. Record your observations in your lab notebook.

9. Cool the resulting clear solution in an ice bath until crystals of alum form. Also, place a small Erlenmeyer flask containing 20 mL of 50% ethanol/water solution into the ice bath to cool for use in step 10.
If no crystals form after 15 - 20 minutes, try scratching the inside of the beaker with a glass stirring rod. The scratching forms small grooves in the glass, and the crystals can adhere to the rough surface. If you are still unsuccessful in obtaining crystals, boil the solution to reduce the volume of liquid by 25% and then cool in the ice bath. When crystals begin to form, leave the beaker in ice for another 10 minutes.

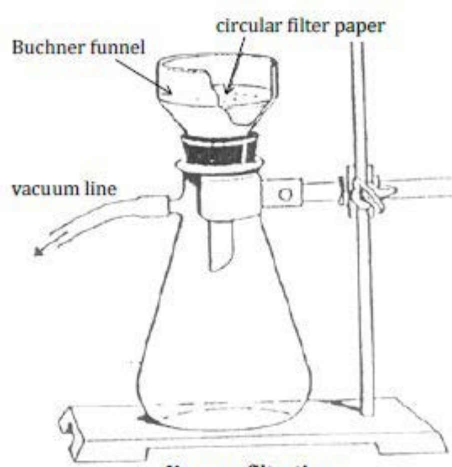
d $Al_2(SO_4)_3(aq) + K_2SO_4(aq) + 24H_2O(l) \longrightarrow 2[KAl(SO_4)_2 \bullet 12H_2O](s)$

This was created in reaction b and is still present in the solution

> **Q:** In reactions a, b, and c, were there any potential ways to lose reactants that are necessary for the final reaction (step d) to form alum?

10. Place a circular filter paper into your Buchner funnel (see setup here). Using vacuum suction, filter and wash the crystals using *ice cold* ethanol/water solution. It's important that the ethanol/water solution is very cold. Continue to apply the vacuum until the filter paper is dry and the crystals do not stick to a stirring rod if touched.

Carefully remove the filter paper from the funnel. Select a clean, empty beaker and weigh it, recording the mass in your lab notebook. Then transfer the crystals into the beaker by scraping them from the paper and record the mass of the beaker plus crystals. Record this in your lab notebook. This is the mass you will use for determining percent yield. [Note: your TA may have additional ideas about drying and transferring the crystals].



Vacuum filtration

Post Lab Question: Please note that there is a post lab question that is part of the lab report (see rubric, "Post Lab Question"). Include the answer to this post lab question right after your analysis, under a separate heading: What is the percent yield of your synthesis of alum? (Provide a brief explanation of your results).

11. **Recrystallization.** The next step is to recrystallize the alum. Recrystallization is a common technique for purifying substances. It can increase purity of the solid because impurities are left in solution. Large crystals often indicate high purity as shown here.

Crystal of alum (source: https://en.wikipedia.org/wiki/Alum, accessed Oct 2016).

Since recrystallization will take time, you will try to grow your crystals in your drawer over the next week. The instructions are shown below. **Your recrystallization results do not have to be included in your lab report. However, if you find that you grew nice crystals and would like to include a picture in your lab report, take a picture with your cell phone (or ask someone else to take a picture for you) and include it in your report.** For this reason, your lab report will be due the next day after you inspect your crystals next week.

Recrystallization Procedure: transfer approximately 5 g of your alum into a 150 mL beaker, add 30 mL distilled water and heat with stirring to dissolve the solid. When all of the alum has dissolved, remove the solution from the heat. Loop some thread over a glass rod and rest the rod across the top of the beaker, to suspend the thread into the solution. Make sure the thread is about ¼" (0.5 cm) below the surface of the liquid but does not touch the sides or bottom of the beaker. Carefully place the solution uncovered in your drawer and allow it to stand until the next laboratory period.

Extra Credit – Extra Challenge (10 pts)

Propose an analogy that could be used to help you make the connection between the macroscopic observations you made during the synthesis of alum and the chemistry occurring at the submicroscopic level. To receive full credit, you should include a statement about the similarities and differences between the analog and target.

Rubric for laboratory reports (due the day after your next lab meeting)

Introduction: **(5 pts)**	**<u>Goal:</u>** To provide a short introduction. **<u>Content:</u>** The summary presents the title of your report, the date the lab work was done, your partner(s) and a couple of overview sentences about what the lab experiment was about.
Data, Results, Evidence: Scientific data that supports the claim. **(25 pts total)**	**<u>Goal:</u>** To describe what you did and what data was collected and observed. **<u>Downloaded Procedure:</u>** Reference the laboratory procedure that was downloaded and the date it was accessed (Synthesis of Alum, InterChemNet, accessed: 10/1/2018). Any changes in procedure should be noted. **<u>Data, Results, and Evidence:</u>** Carefully organize and present the data you collected and any calculations you performed. We recommend that you include chemical equations and explanations that are provided to give some insight into what happens on the atomic scale.
Analysis of Evidence (Reasoning): Scientific explanations that use evidence to construct claims. **(30 pts total)**	**<u>Goal:</u>** To provide the logic to evaluate your data and observations. **<u>Discussion:</u>** Analyze the evidence you will be using to support your claim. It may be helpful to include a discussion of the underlying chemical transformations as part of your evidence at both the molecular and macroscopic scales. Hint: Using your observations in lab and drawings of models representing molecular structure, discuss the interactions of aluminum with other atoms and molecules; including aluminum surrounded by other aluminum atoms, surrounded by solvent molecules, and surrounded by water, potassium and sulfate ions as an alum salt. **<u>Scientific Question:</u>** How do changes in chemical state (solid to solute to crystal) affect the way aluminum interacts with other chemicals? Helpful to include observations about reactions and drawings of aluminum at the atomic scale.
Post Lab Question **(5 pts)**	Include the answer to this post lab question right after your analysis, under a separate heading: What is the percent yield of your synthesis of alum? (Provide a brief explanation of your results).
Claims(s): Statement(s), derived from evidence, using scientific reasoning **(15 pts total)**	**<u>Goal:</u>** To describe what claims or conclusions can be made. **<u>Claims:</u>** Clearly state what claims or conclusions you can make. The logic of your claims builds from the evidence and reasoning presented in the previous sections. Please write your claims clearly in order for them to be assessed reasonably.
Extra Credit **(10 pts total)**	**<u>Goal:</u>** to propose an analogy. Include a discussion of the similarities and differences of the analog and target