Synthesizing Alum
Reaction yields and green chemistry

Introduction:

During this laboratory session, we will depart from an analogy and learning cycle approach. We hope that you will be able to use what you have learned in previous sessions to construct a strong scientific argument based on the experiment that includes evidence, reasoning, and claims.

Goals:

1. To synthesize alum from aluminum soda cans.
2. To make observations of each intermediate chemical reaction in the synthesis.
3. To calculate the theoretical yield, percent yield, and atom economy of the synthesis reaction.
4. To construct a scientific argument about the efficiency of the reaction.

Aluminum cans are often recycled to make more aluminum products, but scrap aluminum metal can also be used to synthesize other useful compounds. Aluminum can be used to produce alum, a chemical that has myriad applications. Some uses of alum are for water purification, making explosives, tanning processes, deodorant sticks, crisping pickles, and medical astringents.

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

\[ 2\text{Al}(s) + 2\text{KOH}(aq) + 4\text{H}_2\text{SO}_4(aq) + 22\text{H}_2\text{O}(l) \rightarrow 2\text{KAl(SO}_4\text{)_2\cdot12\text{H}_2\text{O}(s)} + 3\text{H}_2(g) \]

The name “alum” actually describes several related compounds. The compound that you will synthesize today is potassium aluminum sulfate dodecahydrate \((\text{KAl(SO}_4\text{)_2\cdot12\text{H}_2\text{O})})\). The dot and the 12 \(\text{H}_2\text{O}\) in the formula signal that this is the hydrated form of the compound. When compounds crystallize out of an aqueous solution, water molecules can sometimes fit into the crystalline structure and become bound with the solid. 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.

**Percent yield and theoretical yield:**

With the molar ratios provided by a balanced chemical equation, you can predict the amount of product (in this case, the amount of potassium aluminum sulfate dodecahydrate or alum) 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 that you start with.

However, obtaining the exact theoretical yield in a synthesis reaction is difficult, if not impossible. 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 that can lead to the loss of some of the intermediate compounds necessary for forming the final alum product. The percent yield can be calculated to compare the actual yield and the theoretical yield:

\[
\text{Percent yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100
\]
Reaction efficiency and green chemistry:

**Green chemistry**\(^1\) is an international movement to redesign chemical processes in order to minimize their environmental impact. This is achieved by finding ways to reduce, and in some cases eliminate, environmentally hazardous reactants and products, as well as making sure reactions are as efficient as possible (more products, less waste, and less energy consumption to make those desired products).

There are a variety of ways to maximize the efficiency of a chemical reaction to make it more green. Maximizing the percent yield of a reaction is crucial to optimizing its efficiency. Another important measure of the efficiency of a reaction is its **atom economy**:

\[
\text{Atom economy} = \frac{\text{Mass of desired product}}{\text{Total mass of all reactants}} \times 100
\]

When a reaction is optimized for atom economy, the goal is to maximize the amount of starting materials that end up in the final product, therefore limiting the amount of discarded byproducts that end up as waste. In the perfect chemical process, the mass of desired product and the total mass of the reactants would be equal, resulting in 100% economy.

In today’s experiment, we would like you to consider a host of factors including atom economy, percent yield, energy usage and the possible environmental hazards of reactants as you answer the following question:

**Scientific Question:**
Is the synthesis of potassium aluminum sulfate dodecahydrate ("alum") from aluminum cans an efficient way to recycle aluminum?

There is no wrong or right answer to this question; however, 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.

**Pre-lab Assignment:**

In your lab notebook, please prepare the following information and answer the questions. You must complete the pre-lab before coming to the lab meeting or you will not be permitted to go into the laboratory (questions continue on next page).

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

2. Create a safety information table that includes the chemicals used in the

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\(^1\) You can read more about Green Chemistry on the American Chemical Society’s website: portal.acs.org/portal/PublicWebSite/greenchemistry/index.htm
lab, the hazards associated with them, and any safety handling precautions.

3. 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? (See the example “worked problem” below.)

4. 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 H₂SO₄ would be needed to react according to the equation?

\[
\text{Fe} (s) + \text{H}_2\text{SO}_4(aq) \rightarrow \text{FeSO}_4(aq) + \text{H}_2(g)
\]

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

\[
1.060g \text{ Fe} \times \left( \frac{1.00\ \text{ mole Fe}}{55.845g \text{ Fe}} \right) = 0.0190 \text{ moles Fe}
\]

Step 2. According to the equation for the reaction, for every 1 mole of Fe, we need 1 mole of H₂SO₄. Therefore, we need 0.0190 moles of H₂SO₄.

Step 3. The volume of 1.55M H₂SO₄ solution required is

\[
0.0190 \text{ moles H}_2\text{SO}_4 \times \left( \frac{1000 \text{ mL}}{1.55 \text{ moles}} \right) = 12.3 \text{ mL}
\]

**Laboratory Guide**

On the following pages you will find instructions for doing an experiment. 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.
### 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. Using scissors, cut the strip into small pieces and weigh out about 1 g of the metal. It is not important to have exactly 1.000 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.)

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:** How can you tell that hydrogen gas is produced in this reaction from observing it?

5. When the initial bubbling and foaming has slowed, heat the solution over a Bunsen burner very gently. 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.

\[
2\text{Al(s)} + 2\text{KOH(aq)} + 6\text{H}_2\text{O(l)} + \text{heat} \rightarrow 2\text{KAl(OH)}_4(aq) + 3\text{H}_2(g)
\]
Q: Why can you no longer see the aluminum once the reaction is complete?

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

7. Cool the solution to room temperature and then slowly add 15 mL of 9 M sulfuric acid. 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?

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 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.

Q: In reactions a, b, and c, are there any potential ways to lose reactants that are necessary for the final reaction d to form alum?
10. Vacuum filter and wash the crystals: Collect your crystals by vacuum filtration (see picture to the right for setup). Wash them with the cooled ethanol/water solution. Continue to apply the vacuum to the crystals until the filter paper is dry and the crystals do not stick to a stirring rod if touched. To obtain any product remaining in the funnel and to make sure the crystals are dry, make a paper sandwich by removing the filter paper from the funnel and placing it product side down on top of a piece of paper towel. Press down on the top of the sandwich with another paper towel to remove excess moisture. Transfer the crystals by scraping from the paper to a small beaker (mass previously recorded) and record the mass of the crystals of alum that you have produced. This is the mass you will use for determining percent yield.

Now you will make a larger crystal of alum to observe next lab period (similar to the illustration under the title of the procedure). Recrystallization is a process to purify the alum product. This process results in increased purity because impurities are left in solution. Large crystals often indicate high purity. **This product does not have to be reported in your lab report.**

11. Transfer approximately 5 g of your alum into a 150 mL beaker for recrystallization. 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 it into the solution. Make sure the thread is about 1-2 inches below the surface of the liquid but is not touching the sides or bottom of the beaker. Place the solution uncovered in your drawer and allow it to stand until the next laboratory period.
## Rubric for laboratory reports* (due next lab meeting)

**The purpose of lab reporting** is clear communication of your data and observations, analysis, and claims.

| **Introduction:** (5 pts) | **Goal:** To provide a short introduction.  
**Content:** Provide background information to put the experiment in context. What is alum? What is the percent yield? What is a theoretical yield? What is atom economy? How are they used to assess reaction efficiency? |
|----------------------------|--------------------------------------------------------------------------------------------------|
| **Data, Results, Evidence:**  
Scientific data that supports the claim.  
Submission of the Analog to Target and Designing Experiments Worksheets are required. (30 pts total) | **Goal:** To describe what you did and what data was collected and observed  
**Downloaded Procedure:** Reference the laboratory procedure that was downloaded and the date it was accessed (Alum Synthesis, InterChemNet, accessed: 11/1/2014). Any changes in procedures should be noted.  
**Student Developed Procedure:** The Designing Experiments Worksheet should be included. If insufficient details are present on the worksheet, provide further details in your lab report.  
**Data, Results, and Evidence:** Carefully organize and present the data you collected. Observations can be important data to use in your analysis. Since patterns are often critical to understanding data, present data in Tables as well as Figures. Present the data you collected and the calculations your preformed. What did you observe in each step of the synthesis? Were there opportunities for reactants to be lost along the way? What measures of reaction efficiency did you calculate and how? |
| **Analysis of Evidence (Reasoning):**  
Scientific explanations that use evidence and appropriate chemistry concepts to construct claims. (30 pts total) | **Goal:** To provide the logic to evaluate your data and observations  
**Discussion:** Explain why the evidence you presented supports your claim. What underlying chemical explanations can you provide about your evidence? Use the lab procedure, outside sources, or talk to your instructor to gather information. Be sure to discuss the phenomena at both the submicroscopic (molecular) level and macroscopic (visible to your eyes) level.  
Hints for writing this lab report: Are any patterns evident? Discuss the phenomena at both the submicroscopic (molecular) level & macroscopic (visible to your eyes) level. |
| **Claim(s):**  
Statement(s), derived from evidence, using scientific reasoning. (15 pts total) | **Goal:** To describe what claims or conclusions can be made. What product did you make? What yield did you obtain? What was the atom economy?  
**Claims:** Clearly state what claims or conclusions you can make. The logic of your claims builds from the evidence and reasoning presented in your previous sections. What reasoning can you provide to make meaning of the experiments you conducted (along with outside references). **A good claim will include a short summary of the major pieces of evidence and analysis.** Please write your claims clearly in order for them to be assessed reasonably.  
Hints for writing this lab report: Think about your experimental procedure and how it allows you to understand how to analyze an unknown solution. |

*Lab Course Guidelines and Sample Lab Report, see the general chemistry website for more information