Experiment 3: A First Synthesis Reaction—Preparing Alum from Aluminum

In the last experiment you had the opportunity to deduce and write chemical formulas for some simple ionic compounds. Another goal for this course is to use these formulas to deduce what is happening during a reaction. In this experiment you will investigate a sequence of reactions that begins with Al metal and ends with alum, $K_2SO_4 \bullet Al_2(SO_4)_3 \bullet 24H_2O$, and then identify the type of reactions involved.

Pre-lab Assignment. Read through the laboratory handout and come up with at least one or more questions that will help you complete this experiment. Write your question(s) in the section of your notebook for pre-laboratory work. We will answer these questions at the beginning of lab, so be prepared to share yours! *In addition, bring to lab a sample of aluminum, such as an aluminum can or aluminum foil. You will need approximately 1 gram of aluminum.*

Background. The Brundtland Commission of the United Nations defines sustainable development as meeting our material needs while not limiting the ability of future generations to meet their material needs. A key component of sustainable development is recapturing and reusing the raw materials in products when we no longer need them. This is particularly important when a resource's supply is limited or when obtaining the resource has significant economic, societal, or environmental costs.

Aluminum is one example of a resource where recycling is of great benefit. The major ore of aluminum is bauxite, a mixture of three minerals: gibbsite, or $Al(OH)_3$, boehmite, or γ-AlO(OH), and diaspore, or α–AlO(OH). Although bauxite is readily available—there are sufficient known reserves to meet demand for at least the next 100 years—and although extracting aluminum from bauxite is reasonably inexpensive, the ease and low cost of recycling both extends the lifetime of known reserves and lowers the cost of aluminum; it also limits the real environmental damage associated with mining bauxite. Almost one-third of the aluminum produced in the United States is manufactured from recycled scrap.

In this experiment, you will start with a sample of scrap aluminum and convert it to alum through a sequence of reactions. Alum itself has a variety of use, including in deodorants, paper, water treatment, and "double acting" baking powder.

Procedure. This experiment consists of four steps in which we (1) dissolve aluminum with a strong base, (2) prepare a solution of soluble aluminum sulfate, (3) synthesize alum, and (4) isolate the alum.

The first three steps include several chemical reactions. For each chemical reaction you will find a list of the relevant chemical species; from this list you need to determine which are reactants and which are products and write an appropriate balanced chemical reaction. Finally, you will classify each reaction as one (or as more than one) of the four basic types of chemical reactions:

1. an acid–base reaction, in which a proton is transferred between species
2. a precipitation reaction, in which a solid forms from an exchange of ions between species
3. a complexation reaction, in which one species donates an electron pair to another species
4. an oxidation–reduction reaction, in which electrons are transferred between species

We will briefly review these types of reactions at the beginning of lab.

As you complete each reaction, describe carefully in your lab notebook everything you observe, including the color of solutions and precipitates, whether a gas is produced, and any other noteworthy information. These observations will help you in identifying the reactants and products, and in determining the types of reactions taking place. Note: water (H_2O) is present in all reaction mixtures, so you may include it as a reactant or a product as needed to balance a reaction.

Step 1. Dissolve Aluminum with a Strong Base

This step is a single reaction, for which the relevant species are H_2, $Al(OH)_4^-$, Al, KOH, and K^+.

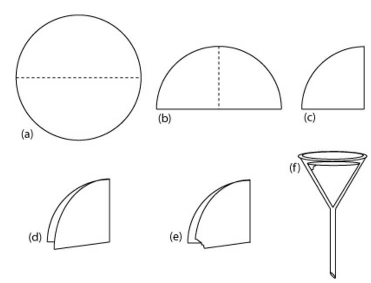
> ***Caution: much of this step is completed in a hood. Once you begin working in the hood, do not remove your beaker until the directions specifically note that you can do so.***

Weigh an approximately 1-g piece of scrap aluminum. If your sample is too large (>1.1 g), then trim as needed; if it is too small (<0.9 g), then add additional material. Cut your sample of Al into small pieces, reweigh to determine the sample's exact mass, and then place the sample in a 250 mL beaker.

Working in a hood, add 15 mL of 6 M potassium hydroxide and cover the beaker with a watch glass. Place the beaker on a hot plate and heat gradually using a moderate setting (do not turn the heat up all the way!). Watch the solution carefully and as soon as it starts to react visibly, add 35 mL of deionized H_2O. Note the approximate volume of solution in your beaker after this addition, and add water to maintain this volume while it heats. Do not add excess water as this dilutes the base and slows the reaction.

Continue heating until the aluminum metal is no longer visible, and the reaction ceases. When you think the reaction is complete, carefully remove the beaker from the hot plate—use a heat resistant glove or paper towels to protect your hands—and gently swirl the solution; if foam appears on the surface of the liquid, then the reaction is not yet complete and needs additional heating. You may observe fine black particles or a plastic film suspended in the solution; the former is carbon, which is sometimes included in manufactured aluminum and the latter is a coating on your sample of aluminum. These are removed by filtration.

You may now return to your lab bench.



Using the illustration to the right as a guideline, prepare a piece of filter paper by folding it into quarters (a-e) and placing it in your short stem funnel (f). Wet the paper with a small amount of distilled water to ensure a good seal at the top. Place the funnel over a 250 mL Erlenmeyer flask, which will serve as a vessel for collecting your filtrate. While your solution of dissolved aluminum is still hot, pass it through the filter paper to collect the filtrate. Rinse your reaction vessel with two portions of approximately 3 mL of deionized water each, and pass these through the filter paper as well. Discard the filter paper and save the filtrate for the next step.

Step 2. Prepare a Solution of Soluble Aluminum Sulfate

This step consists of two reactions. For the first reaction, the relevant species are $Al(OH)_3$, H_2SO_4, $Al(OH)_4^-$, and SO_4^{2-};for the second reaction they are $Al_2(SO_4)_3$, $Al(OH)_3$, and H_2SO_4.

Place 35 mL of 6 M sulfuric acid in a small flask. Slowly add the sulfuric acid to the filtrate from step one until you observe the formation of an insoluble precipitate. Place the beaker on a hot plate and heat the solution while adding the rest of your sulfuric acid. Allow the solution to warm until it just starts to steam. If your solid fails to dissolve completely, add a little more acid (2 mL) and heat a bit longer. Repeat this process until the solid dissolves completely. Be sure to record the total amount of acid you added to your flask. Save this solution for the next step.

Step 3. Synthesize Alum

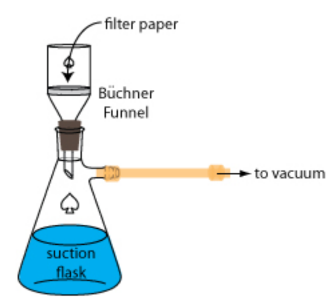
This step consists of a single reaction, for which the relevant species are K_2SO_4, $Al_2(SO_4)_3$, and $K_2SO_4 \bullet Al_2(SO_4)_3 \bullet 24H_2O$.

Using ice and water, prepare an ice bath in a container large enough to hold the beaker holding your reaction mixture. Place your beaker in the ice bath and allow the solution to cool. Crystals of alum will appear on the bottom of your beaker as a fine, white powder. If crystals do not form after approximately 10-15 minutes, scratch the bottom of the beaker with a glass stirring rod or add a seed crystal of alum from someone else's product to aid the crystal formation.

(Note: While you are cooling your solution, place 10 mL of methanol in a small flask and begin cooling it in the ice bath for use in the next step.)

Step 4. Isolate Your Alum

To isolate your alum, set up on the vacuum filtration apparatus illustrated below. Weigh your filter paper before placing it in the Büchner funnel. Check with your instructor before proceeding to make sure you correctly set up the vacuum filtration apparatus.



Transfer your alum crystals by swirling the liquid and crystals in your beaker and pouring the resulting suspension into the Büchner funnel while applying suction with a vacuum aspirator. Use a spatula to transfer as much as possible of your remaining crystals. To complete the transfer of your crystals to the filter, you will use some of the filtrate in your suction flask. Break the vacuum by removing the hose from the side arm of your flask. Remove the Büchner funnel and pour the filtrate into a small clean beaker. Replace the Büchner funnel, reattach the hose, and reapply the vacuum. Using small portions of your filtrate, transfer the remaining alum crystals from the reaction vessel into the Büchner funnel. Finally, wash the crystals in the Büchner funnel using several small 2 mL portions of your filtrate.

Caution: do not use deionized water to transfer or wash your crystals as it will dissolve your alum.

Your alum is still a bit wet at this point. To remove excess water from your crystals, use a disposable pipet to add slowly the 10 mL of cold methanol prepared during the previous step. Note: when the methanol hits the filtrate in the filtration flask, it will form crystals of excess K_2SO_4, not alum; do not attempt to collect these crystals.

Remove the top part of your Büchner funnel—you can pop it off from the remainder of the funnel—and allow the alum to dry in your drawer until the next lab period. Transfer the filter paper and your crystals to a watchglass and determine the mass of your alum crystals (be sure to correct for the mass of your filter paper). Transfer your crystals to a glass vial and label it with your name, the formula of alum, and the mass of alum.

Waste Disposal. Dispose of all waste into the container provided in the hood.

Lab Report. Complete the worksheet provided by your instructor.

References. This laboratory is adapted from one developed by Deborah Simon of Whitman College.