Laboratory 2: Recycling Aluminum: Preparation of Alum from Scrap Aluminum

Background Information

Starting a couple of years ago, DePauw University has been investigating ways to implement sustainability practices and to lessen its impact on the environment. One key component in preserving our resources for future generations is to reuse our raw materials over and over again after they’ve reach the end of their usefulness in their present form. In this week’s lab you will take scrap aluminum (in the form of aluminum cans, foil, etc.) and convert it to a useful compound called alum. We will utilize the alum later in the semester to make a form of aluminum called anodized aluminum that can be dyed different colors and is found in many everyday products such as a water bottle clips and flashlights. Alum itself is also used in deodorant, paper, water treatment, and “double acting” baking powder.

One of the goals of this course is to learn to convert what you are seeing when you perform steps the lab into balanced chemical equations. In this experiment we will be investigating some of the reactions that aluminum can undergo, starting with Al metal and ending with alum, K$_2$SO$_4$· Al$_2$(SO$_4$)$_3$·24 H$_2$O (a pretty crazy formula compared to the others we've seen!) For each of the reactions involved, we can write balanced chemical reactions, showing the relationships between moles of reactants and moles of products, related by small whole number coefficients (mole ratios).

In this experiment, you will start with a piece of scrap aluminum metal, transform it sequentially into a variety of aluminum compounds, and finally recover alum at the end of the experiment. At the end, you will test your alum to determine what percentage of its weight is composed of hydrate water molecules.

You will observe each of the reactions carefully and record your observations in your notebook, trying to figure out whether particular chemicals are on the product or reactant side of the reaction, and writing a balanced equation. You will also have to identify states of matter for the various chemicals (so watch closely!). You will be trying to classify the reactions that you observe according to the types of reactions we will be discussing in class:

1. redox: reactions where electrons are transferred between reagents
2. precipitation: reactions where a solid forms from exchange of ions
3. acid-base: reactions where a proton is transferred
4. complexation: where a Lewis acid-base reaction occurs (electron pair donation)

In some cases more than one type of reaction is occurring.

Pre-lab Assignment

1. Read the entire laboratory and come up with a chemical question that you have. Write the question in your prelab/introduction section of your lab notebook and be prepared to share with the class.
2. Find some aluminum to convert to alum. Look in the campus recycle bins for an aluminum can or some aluminum foil. You will only need 1 gram of aluminum so multiple people can share a can for instance. Each person will do this lab individually, so each person will need enough aluminum to do his or her own experiment.

Experimental Procedure

As you go through this experiment, always be thinking ahead to the next step of the reaction. What reagents or equipment will you need to carry out the next step? There is a lot of waiting as the different reactions occur, so take advantage of the time by gathering together equipment for the next step (you will finish more quickly that way!). As you carry out the experiment, describe all chemical reactions in your notebooks, including the color of solutions and precipitates, gases produced and other noteworthy information. For each reaction you will eventually have to write a chemical reaction for the process occurring. In some cases you will have to use the information and hints given as well as your observations to determine the reactions occurring. For each step, the chemicals necessary to write a balanced equation are given, but the products and reactants are not designated. IMPORTANT NOTE: in any step in solution, water (H$_2$O) is present and may be required as an additional reagent to balance the equation, but it is only included in a reaction if it changes over the course of the reaction.

Step 1. Dissolution of Aluminum in Strong Base

*Relevant Chemicals: H$_2$, Al(OH)$_4^-$, Al, KOH, K$^+$*

*This step must be done in a hood. Do not leave your beaker out on the counter or carry it around the lab after you have added 6 M potassium hydroxide to it. Only remove it from the hood after the directions specifically note you can do so.*

Weigh a 1 gram (+/- 0.1 g) piece of scrap aluminum, recording the weight to the nearest milligram. Use the tin snips to cut it to the required size, or add another scrap. It doesn’t have to be exact, just be sure to record the actual mass. Cut the Al into small pieces, then place it in a 250 mL beaker.

**IN THE HOOD:** Add 15 mL 6 M potassium hydroxide solution. Cover the beaker with a watch glass.

Place the beaker on a hot plate and heat gradually starting at a moderate setting (do not turn the heat up all the way!). Watch the solution carefully and as soon as it starts to visibly react, add 35 mL deionized H$_2$O. Note the volume of the liquid in your beaker after the addition, and use your water wash bottle to maintain this height while it heats. **Do not add excess water** and dilute the base too much.
Continue heating until the metal is no longer visible, and the reaction has stopped. Check this by carefully removing the beaker from the hot plate (using a heat resistant glove or paper towels to protect your hands), placing it on a white sheet of paper, and gently swirling it. If foam appears on the surface of the liquid, it is still reacting, and needs to be heated longer. There may be fine black particles suspended in the liquid at this point; this is a contaminant coating from the scrap metal piece, and is not part of the reaction—it is OK if all the solid does not completely dissolve. **WHEN THE REACTION IS FINISHED YOU CAN REMOVE IT FROM THE HOOD AND CONTINUE.**

Prepare a piece of large filter paper by folding it into quarters. Insert the paper wedge into your short stem funnel and separate the layers so that the filter paper completely covers the inside of the funnel (three layers will be on one side and one on the other). Wet the paper with a small amount of distilled water (this is to prevent the solid from going over the side of the paper and into the liquid below!). Place the funnel over a 250 mL beaker that will serve as a collection vessel for your filtrate. While the liquid is still hot, pour it into the funnel to filter the hot liquid. Rinse the reaction vessel with two portions of about 3 mL of deionized water and pour that through the filter as well.

**Step 2-4. Addition of Sulfuric Acid**

The addition of sulfuric acid actually leads to three separate reactions, the first two of which happen so quickly that it is hard to distinguish them!

2. **Relevant Chemicals: H$_2$SO$_4$, K$_2$SO$_4$, KOH**

Measure out 35 mL 6 M sulfuric acid into a small flask. The very first bit of acid that is added is reacting with excess base that is left in the beaker from the previous reaction.

3. **Relevant Chemicals: Al(OH)$_3$, H$_2$SO$_4$, Al(OH)$_4^-$, SO$_4^{2-}$**

Continue to add the acid slowly until you see an insoluble precipitate form.

4. **Relevant Chemicals: Al(OH)$_3$, Al$_2$(SO$_4$)$_3$, H$_2$SO$_4$**

Now place the beaker on a hot plate and heat the solution while adding the rest of the sulfuric acid. Allow the solution to warm until it just starts to steam. If your solid fails to completely dissolve after adding all of the acid and allowing it to react for a few minutes, add a little more acid (2 mL) and heat a bit longer. Repeat this process until the solid is completely dissolved. Be sure to record the total amount of acid added to your flask.

**Step 5. Relevant Chemicals: K$_2$SO$_4$, K$_2$SO$_4$•Al$_2$(SO$_4$)$_3$•24H$_2$O, Al$_2$(SO$_4$)$_3$**

When this solution, which contains two soluble salts is cooled, they link up to form a double salt, which crystallizes and precipitates from solution. This double salt is called "alum."
Make an ice bath in a 600 or 800 mL beaker with water and ice. Place your reaction vessel in the ice bath, and allow the solution to drop to 5°C or lower to let the alum crystallize. The crystals will appear on the bottom of your beaker as a very fine, sandy, white layer. If crystals do not form after about 10 minutes at the correct temperature, scratch the bottom of the beaker with a glass stirring rod or add a seed crystal from someone else’s product to aid the crystal formation.

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

You will next quantitatively transfer (meaning do your best to get all of your solid WITHOUT re-dissolving your crystals) the alum to a Buchner funnel set up on a vacuum apparatus as shown. You may want to weigh your filter paper before using in order to subtract its weight from your alum at the end. Set up the filtration apparatus and check with your instructor before proceeding to make sure you have set it up correctly before transferring the crystals.

To transfer the crystals, a good strategy is to swirl the liquid and crystals in the beaker, then pour to dislodge crystals on the bottom of the beaker. Pour the liquid into the Buchner funnel, and then use a spatula to transfer some of the remaining of the crystals. **Do not add any pure water to wash the crystals or to help transfer the last remaining crystals at this point or you will dissolve your alum!** Instead, once you have drawn the liquid off the filtered crystals, break the vacuum by removing the hose on the side arm of your flask, open the suction flask and pour the filtrate (liquid) into a small clean beaker. Replace the filter flask and resume the vacuum. Now use the some of the filtrate (only as much as you need!) to wash the remaining alum crystals from the reaction vessel into the filter and then wash the crystals themselves, using several small rinses of about 2 mL each.

Your alum is still a bit wet at this point. Wash your crystals free of excess water by slowly rinsing the crystals with 10 mL of **COLD** methanol using a disposable pipette. **(NOTE: When the methanol hits the filtrate in the filtration flask, it will form crystals of excess K₂SO₄, not alum! **Discard this filtrate, and do not pour it back over the alum!**)**

**Transfer the Alum and Allow it to Dry**
Quantitatively transfer the alum (again do your best to get all of your solid) into a watch glass or simply let it dry in the top section of you Büchner funnel (you can pop it off the funnel part!). Allow it to dry in your desk for at least two days, then come in and obtain its mass. Use your spatula to gently scrape the alum free from the filter paper before weighing. When you have obtained the mass, place it in a glass vial labeled with your
name, the formula, and the mass. If you weighed your filter paper before filtering you can just weigh your alum and filter paper and subtract to get your mass of alum. You can also weigh an empty container, scrape the alum into the container and weigh again to obtain your mass of alum by subtracting the two values.

**Mass Percent Water of Alum Sample**
Since alum is a hydrated salt, the water molecules that are attached to the salt contribute to its overall mass. We can determine how much water is part of the structure of the crystal (hydrate water) by heating the alum in an oven at a higher temperature and seeing how much mass is lost. Take about 1 g (+/- 200 mg) of your alum and weigh it and record the mass in your notebook. Place it in a small beaker. Label your beaker without using paper (it will char!) and place it in the oven set to 110 °C. After 48 hours, remove your sample using the heat resistant gloves provided, allow the sample to cool to room temperature, and reweigh your sample to see how much of the weight has been lost. When weighing your sample be sure to tare the balance or obtain the mass of the weighing vessel prior to putting the solid into it. In your notebook make a note of any differences in the appearance of the solid before and after heating. Save both your undried and dried alum for inspection by the lab instructor during the next lab period.

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

**Lab Report**
Complete the worksheet provided by your instructor and attach to your lab book pages when submitting your lab.

**References**
This laboratory is adapted from a version developed by Deberah Simon of Whitman College.