CHM115 Lab 2

Stoichiometry – Synthesis of Alum

Purpose: In this lab you are going to carry out a series of reactions to transform a piece of aluminum into white crystals (alum). You will use stoichiometry to figure out the limiting reagents and yield for your reaction.

Background: Aluminum is the third most common element in the earth's crust, but it forms very stable bonds. It used to be very difficult to isolate, so pure aluminum was worth more than gold. We owe cheap aluminum to Charles Hall, who began experimenting with it when he was an undergraduate researcher at Oberlin College, and finally figured out how to do it when he was 22.[1] In this lab, you are going to do the opposite of Hall's experiment, turning metallic aluminum back into a naturally occurring type of salt called alum. Alums are a class of salts that have sulfate (SO_4^{2-}) as the anion and two different cations, one of which is often (but not always) aluminum (Al^{3+}). They have been used since at least 1500 BCE for a variety of purposes including styptics (to stopping bleeding), aftershaves, water purification systems, flame retardants, anti-bacterial agents and baking powder ingredients. The specific alum you will make is potassium aluminum sulfate dodecahydrate [$KAl(SO_4)_2 \cdot 12H_2O$] which is sold today in "natural" crystal deodorants. Please do not use your product from lab as deodorant because a) it might not be pure and the impurities could be very hazardous to your skin and b) it doesn't work very well and you will smell.

In carrying out these reactions, stoichiometry will determine how much reactant is consumed and how much product is formed. Some sections will be using aluminum cans and some will be using aluminum foil. Check with your instructor to see if you need to bring an aluminum can. Do not get shards of metal near the balances. There is lots of waiting around during this lab, you can do the "Prepare for next week's lab" at any point, and there are lots of calculations to work on.

Procedure – This is the part you write in your notebook. You will work alone for this lab.

Reaction 1: $2\ Al(s) + 2\ KOH\ (aq) + 6\ H_2O \longrightarrow 2\ KAl(OH)_4(aq) + 3\ H_2(g)$

1. If using a can: Cut a piece out of your can that is the same size as the cardboard template. Deposit extra scraps in the box. Scour both sides of your can thoroughly to remove all traces of paint and plastic coating. You can't see the plastic, just go over the entire surface completely. Wipe surface clean with a paper towel.
   If using foil: Cut a piece the same size as the template. Wipe surface clean with a paper towel.
2. Use the top-loading balance to weigh your piece of aluminum. It should be 0.50 +/- 0.1 g. Adjust the amount of metal to get it within this range. Do not get scraps of metal near the balance. Record the mass.

[1] For the complete story (and lots of other history of chemistry), go to the American Chemical Society National Historic Chemical Landmarks.
http://acswebcontent.acs.org/education/chemical_landmarks/timeline/timeline2/timeline2.html

3. Use the scissors to cut the metal into bits about the size of a pencil eraser. Place the bits into a clean 250 mL beaker.
4. Using gloves, obtain 25 mL of 1.4 M KOH solution from the hood in a graduated cylinder. Set the cylinder in a beaker to avoid spills. Record the exact volume. KOH is a strong base and will dissolve skin. If exposed, rinse for 5 minutes with cold water.
5. Set up a tripod (check the legs!), wire gauze, and your beaker with the metal. Position the fume extractor over the top of the beaker. Add the KOH solution to the aluminum.
6. Light the Bunsen burner and heat the solution until all the aluminum is dissolved. Do not boil it. You should, however, see bubbles forming. What are these bubbles? (hint: look at the balanced reaction).
7. While heating, take a piece of filter paper, carefully fold it in fourths and lay it in the glass funnel from the stock room, as demonstrated by your instructor. Place the funnel in a 250 mL Erlenmeyer flask When no more bubbles form, turn off the Bunsen burner. You should have a solution of potassium aluminum hydroxide plus any impurities that were in the aluminum. You need to filter to remove the impurities. Carefully pour the solution through the filter paper, making sure to stay below the top of the paper. You should have a colorless solution, although it's ok if a slight gray color comes through. If there is solid, you must have holes in your filtration set up and you need to repeat this step.

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

8. With a clean beaker, wear gloves to obtain 10 mL of 9.0 M H_2SO_4. Set the beaker in a weigh boat to prevent it from contaminating the bench top. If you spill any acid, clean it immediately up with sopping wet (NOT dry) paper towels. When the $KAl(OH)_4$ solution is cool, slowly add some of the sulfuric acid while swirling. Gelatinous aluminum hydroxide will form. This reaction gives off heat, be careful.

Reaction 3: $2\ Al(OH)_3(s) + 3\ H_2SO_4\ (aq) \longrightarrow Al_2(SO_4)_3(aq) + 6\ H_2O$

9. As you continue adding the acid, another reaction will happen in which the white aluminum hydroxide becomes aluminum sulfate and dissolves. If there are any white flecks left in the solution after the addition of the H_2SO_4, place the flask on the Bunsen burner apparatus and heat gently until they dissolve.

Reaction 4: $Al_2(SO_4)_3(aq) + K_2SO_4(aq) + 24\ H_2O \longrightarrow 2\ (KAl(SO_4)_2 \cdot 12\ H_2O)\ (s)$

10. The ions to form alum are now in solution. To get them out, you need to cool the reaction. Make an ice water bath and allow the reaction to cool for ~ 5 minutes. Be sure the flask doesn't tip over as the ice melts.

11. If crystals have started forming, great. If not, add 1 “seed” crystal from the container. Let sit for ~ 15 more minutes.
12. While waiting, obtain ~ 5 mL of alcohol/water mixture and cool it in the ice bath. Make sure your vacuum filtration setup is clean, with a Buchner funnel, filter flask and aspirator.
13. Place a filter paper disk flat in the Buchner funnel. Turn on the aspirator. Swirl your crystals and pour into the Buchner funnel. Scrape any remaining crystals out with the rubber policeman. When there is no more water coming through the filter, use the cold alcohol to rinse out any material remaining in the Erlenmeyer. Pour over the crystals. Let air suck through the filter for 5 minutes to aid in drying.
14. Scrape as many crystals as you can onto a watch glass out of the Buchner funnel and off the filter paper disk. Weigh the dry crystals.

Data Table
Mass of Aluminum ________________
Volume of 1.4 M KOH ____________
Volume of 9 M H_2SO_4 ______________
Mass of alum ______________

Make the unknown concentration NaOH solution for the next lab.
Each person should make up one bottle of solution.

1. Take a 1 L brown bottle from the bottle supply. Be sure it is labeled with your name, drawer number, lab day and time and ~0.13 M NaOH solution.
2. Measure 130 mL of 1 M NaOH into a clean beaker. Pour it into the bottle.
3. Fill the bottle almost to the top with deionized water. Cap the bottle and shake the solution carefully.
4. Put the bottle in your drawer for use next week.