Chem 210, Winona State University, Fall 2022

EXPERIMENT 3

Chemical Reactions and Mole Calculations: Synthesis of Alum from Scrap Aluminum

Materials Needed

aluminum scraps from a beverage can, glass wool, 4 M KOH(aq), 9 M H_2SO_4, Büchner funnel, filter paper, filter flask, beakers, hot plate/stirrer, magnetic stirbar

Relevant Textbook Reading

Smith, Chapter 5.5-5.9

Background

In this experiment you will convert scrap aluminum from a used soda can into a useful aluminum compound, aluminum potassium sulfate dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$, commonly known as alum. Alum currently sells for approx. $20 per kg and is used extensively in the dye industry. It is also used in glues and cements, baking powders, explosives, water purification, and as a hardening agent in microscopy. It is available in the spices section of most grocery stores because of its usefulness for pickling and canning.

This experiment demonstrates that waste material can often be converted to valuable substances as a means of recycling it. More importantly, it will provide you with experience in the types of procedures that are used to carry out chemical reactions in a lab and provide an example of how mole calculations are used to determine the efficiency of a reaction process, i.e., the percent yield. You might be surprised at how much alum is afforded from the small mass of aluminum that is started with. Do note that complete efficiency in a chemical process (i.e., 100% yield) is only very rarely achieved and that the yield you obtain will probably be quite a bit lower than 100%. Factors such as reactant purity, reaction rates, equilibrium constants, undesired side reactions, and physical losses during transfers can all lower the % yield obtained.

The experiment will afford you experience some standard laboratory techniques including volume measurements of liquids, filtration, stirring, and recrystallization.

The conversion of aluminum to alum involves the following reactions carried out in sequence

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

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

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

(4) $K^+(aq) + Al^{3+}(aq) + 2\,SO_4^{2-}(aq) + 12\,H_2O(l) \longrightarrow KAl(SO_4)_2 \cdot 12H_2O(s)$ (alum)

The overall equation is:

$$2\,Al + 2\,KOH + 4\,H_2SO_4 + 22\,H_2O \longrightarrow 2\,KAl(SO_4)_2 \cdot 12H_2O + 3\,H_2$$

Procedures

1. Use steel wool to remove as much paint as possible from a piece of scrap aluminum. The inside of the can is protected with a plastic coating and you should remove this also. Weigh the cleaned strip of aluminum to the nearest 0.001 grams.

2. Cut the Al sample into small squares and place the squares in a clean 100-mL or 150-mL beaker.

SAFETY: Perform all of the following steps inside your assigned fume hood. Both KOH and sulfuric acid solutions are extremely corrosive so wear gloves and avoid skin contact with the solutions being used!

3. Reaction #1. Carefully add 20 mL of 4 M potassium hydroxide, KOH. Bubbles of hydrogen gas should evolve. Place your beaker on a hot plate (set on low) to speed up the reaction. When hydrogen bubbles are no longer formed, the reaction is complete. This should take 10-15 min. Remove the beaker from the hot plate and set it aside in the fume hood.

4. Set up a 250-mL filter flask and Büchner funnel as demonstrated by the instructor. Also see the part about Vacuum Filtration at https://orgchemboulder.com/Technique/Procedures/Filtration/Filtration.shtml. Don't forget to clamp the filter flask to a ring stand. If the solution is still warm, cool it by placing the beaker in an ice bath for a few minutes. Turn on the vacuum and pour the solution through slowly. Rinse the beaker two times with small portions (<5 mL) of distilled water, pouring each rinse through the filter. Transfer the clear colorless filtrate to a clean 250-mL beaker.

5. Reactions #2 and 3. To the filtrate, slowly and carefully, with stirring, add 15 mL of 6 M sulfuric acid. White lumps of $Al(OH)_3$ should form in the solution. Place the flask on a hot plate and heat and stir the mixture to dissolve the white lumps. (This might take 10 min) Excess sulfuric acid may be added dropwise, but no more than 30 mL total, in order to totally dissolve the $Al(OH)_3$, and give a clear solution. (Adding the excess acid dilutes the sample and makes it more difficult to crystallize the product, so be cautious when adding excess acid.)

6. “Reaction” #4. Cool the clear solution in an ice bath for at least 15 min. Crystals of alum should fall to the bottom of the beaker. If no crystals form, scratch the bottom of the beaker with your stirring rod. If that fails, add a "seed" crystal of alum to facilitate crystallization.

7. While the solution is cooling, reassemble the filtration apparatus. Be sure to weigh the filter paper at this point. Slowly filter the solution containing the crystals. Rinse the beaker once with 5 mL of cool distilled water and once with 5 mL of isopropyl (rubbing) alcohol, pouring each rinse through the filter. Allow the aspirator to pull air through the crystals until they appear dry, at least 5 minutes. Remove the filter from the flask. More crystals may form in the filtered solution (filtrate). If you have time, filter these crystals as just described, and add these to the first crystal crop.

8. Obtain a clean, dry beaker. Find and record the mass of this empty beaker. Remove the damp crystals and filter paper from the filter and place them into the pre-weighed beaker. Place the beaker containing your crystals in your locker until next week.

9. Weigh the filter and crystals, and determine the mass of alum obtained. Calculate the percent yield.

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PRE-LABORATORY QUESTIONS

Name ____Section ______ Date ______

1. Calculate the molar mass of alum. (The formula is given in the introduction.) Show your calculation.

2. If 0.50 g of aluminum is started with and 5.0 g of alum are obtained at the end of the experiment, what is the percent yield. Assume that aluminum is the limiting reagent.

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IN-LAB OBSERVATIONS/DATA

Names ____Section ______ Date ______

DATA/OBSERVATIONS

Mass of aluminum scrap used, g

Appearance of aluminum scrap

Appearance of reacting aluminum/KOH solution

Appearance of solution before filtration

Appearance of solution after filtration

Approximate time allowed for crystal formation, min

Appearance of filtrate for 2^{nd} filtration

Appearance of damp alum crystals (week 1)

Mass of damp alum crystals, g (week 1)

Appearance of dry alum crystals (week 2)

Mass of dry alum crystals, g (week 2)

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REPORT SHEET

Names ____Section ______ Date ______

Results

	Mass (g)	**Moles**	**Percent of Theoretical**
Scrap Al reactant			**Not applicable**
Alum product yield (wet)			
Alum product yield (dry)			

Questions

1. Show your calculation of the % yield of alum for your experiment.

2. List and explain three major factors that explain why a l00% yield was not obtained. Be specific. See the introduction of this handout.