Experiment 7. Synthesis of Aluminum Sulfate Dodecahydrate (Alum)

INTRODUCTION

In this experiment, we will Synthesize Potassium Aluminum Sulfate dodecahydrate, crystallize the compound, and test for purity, using a melting point measurement.

BACKGROUND

This lab makes use of a number of important wet chemistry synthesis techniques. It's like following a recipe to make a cake. When you follow the recipe, you have a cake. Forget the baking soda or the eggs, you have a solid lump. In this lab, you will attempt to produce crystals of Aluminum Sulfate dodecahydrate. If you carefully follow instructions, you should be able to produce the crystals. Miss a few key steps and the results won't be there. This lab demonstrates stoichiometry and measurement of the percentage yield. Several new techniques are introduced, such as use of a vacuum funnel and measuring the melting point of a compound, as a way to determine purity.

An alum is a hydrated double sulfate salt with the general formula $M^{+}M^{3+}(SO_4)_2{\cdot}12H_2O$.
M^{+} is a univalent cation, commonly Na^{+},K^{+}, Tl^{+}, NH_4^{+}, or Ag^{+}; M^{3+} is a trivalent cation, commonly Al^{3+}, Fe^{3+}, Cr^{3+}, Ti^{3+}, or Co^{3+}. Several common alums, and their uses are detailed below. This experiment describes the preparation of potassium alum, or Potassium Aluminum Sulfate dodecahydrate.

Name	***Formula***	***Uses***
Sodium aluminum sulfate dodecahydrate	$NaAl(SO_4)_2{\cdot}12H_2O$	Baking powers; hydrolysis of Al^{3+} releases H+ in water to react with the HCO_3^- in baking soda to produce CO_2, causing the dough to rise
Potassium aluminum sulfate dodecahydrate	$KAl(SO_4)_2{\cdot}12H_2O$	Water purification, sewage treatment, and fire extinguishers
Ammonium aluminum Sulfate dodecahydrate	$NH_4Al(SO_4)_2{\cdot}12H_2O$	Pickling cucumbers
Potassium chromium (III) sulfate dodecahydrate	$KCr(SO_4)_2{\cdot}12H_2O$	Tanning leather and waterproofing fabrics
Ammonium ferric sulfate dodecahydrate	$NH_4Fe(SO_4)_2{\cdot}12H_2O$	Mordant in dying and printing textiles (makes colors more intense)

Potassium Aluminum Sulfate dodecahydrate, $KAl(SO_4)_2{\cdot}12H_2O$, is prepared from aluminum and standard chemicals found in the general chemistry lab. We set up the experiment so that aluminum is the limiting reagent. Aluminum metal rapidly reacts with hot aqueous KOH producing a soluble potassium aluminate salt solution.

$$2\ Al_{(s)} + 2\ KOH_{(aq)} + 6\ H_2O_{(l)} \rightarrow 2\ K^{+}_{(aq)} + 2\ Al(OH)_4^{-}{}_{(aq)} + 3\ H_{2(g)}$$

When treated with sulfuric acid, the aluminate ion, $Al(OH)_4^{-}{}_{(aq)}$ precipitates as aluminum hydroxide $Al(OH)_3$, which further reacts and dissolves with the application of heat.

$$Al(OH)_4^{-}{}_{(aq)} + H^{+} \rightarrow Al(OH)_{3(s)}$$

$$Al(OH)_{3(s)} + 3H^{+} \rightarrow Al^{3+} + 3\ H_2O$$

There are a number of other “spectator ions” dissolved in the solutions above, such as potassium and sulfate. We removed them from these equations so that the net ionic equations were easy to see. These spectators now play a part in making the final compound, which crystallizes out as octahedral-shaped crystals when the nearly saturated solution cools.

$$K^+_{(aq)} + Al^{3+} + 2\ SO_4^{2-} + 12\ H_2O \rightarrow KAl(SO_4)_2{\cdot}12H_2O$$

When the above 4 steps are considered together, the ***overall*** reaction for the synthesis of potassium alum is:

$$2\ KOH_{(aq)} + 2\ Al_{(s)} + 4\ H_2SO_4 + 10\ H_2O \rightarrow 2\ KAl(SO_4)_2{\cdot}12H_2O_{(s)} + 3\ H_{2(aq)}$$

Purity calculations: The melting point of a mixture is a function of the molality of the solute in the solvent, a concept studied at the end of the semester. In this case “solute” is the impurity in the desired compound. The melting point of a compound is reduced in proportional to the amount of impurity contained. If the melting point is significantly lower than the melting point of the control sample, we can tell that it’s very impure. It’s always good to compare a melting point against a “known good sample”, to avoid problems from uncalibrated thermometers.

PROCEDURE

Overview: A known mass of aluminum is used to synthesize potassium aluminum sulfate dodecahydrate by reactions with potassium hydroxide and sulfuric acid. The theoretical yield can be determined by stoichiometry. The percent yield can then be determined. The purity is checked by comparing the melting point to the melting point of a "pure" sample.

Caution: You are working with strong base and strong acids. Wear goggles and avoid skin contact.

Part A: Generate the $Al(OH)_4^-{}_{(aq)}$

1. Cut about 0.5 grams aluminum foil into small pieces and mass it, recording three significant figures.
2. Place the aluminum into a 150 ml beaker and add 25 mL of 2 M KOH.
3. Heat the beaker on a hot plate IN THE HOOD. Warm the beaker gently to initiate the reaction. Pull the beaker off the hot plate when/if it boils. As the reaction proceeds, hydrogen gas is evolved, as is evidenced by the "fizzing" at the edges of the aluminum pieces.
4. When no further reaction is evident and the aluminum is dissolved, gravity filter the warm solution through a small cotton plug (rather than filter paper) to remove the insoluble impurities. If evidence of solid particles appears in the filtrate, repeat the filtration procedure.
    a. The cotton plug should have a diameter of about 1 cm. It rests in the bottom of the funnel. A large piece of cotton absorbs too much of your solution (net: poor yield).

Part B: Formation of aluminum hydroxide

1. Allow the clear solution (the filtrate) to cool in a 150 mL beaker.
2. While stirring, slowly add about 8 mL of the 6 M sulfuric acid. Insoluble $Al(OH)_3$ forms.
3. Dissolve the aluminum hydroxide by gently heating the reaction mixture with stirring until the aluminum hydroxide dissolves.
4. Dropwise continue to add just enough 6 M sulfuric acid to cause the solution to go clear (3-8 mL more). The solution can boil, but does not have to.
5. Record the total amount of sulfuric acid added.

Part C: Crystallization of Alum

1. Remove the solution from the heat; filter only if solids are present. Put a watch glass over the beaker and let it sit. If no crystals have started after 30 minutes, gently scratch the bottom of the beaker. Put the beaker in your drawer until the next class session, when you will continue. Slow cooling yields the largest (purest) crystals with the greatest yield.
    a. If only one lab is allocated to this lab, cool the solution in an ice bath. Alum crystals should form within 30 minutes. If crystals do not form, gently heat to reduce the volume by one-half and recool for 20 minutes.

Part D: Isolate and wash the alum crystals

1. Obtain the mass of the filter paper and watch glass together.
2. Vacuum filter the alum crystals from the solution.
3. Wash the crystals on the filter paper with two 5-mL portions of a 50% (by volume) ethanol-water solution. Maintain the suction until the crystals appear dry.
4. Transfer the filter paper and crystals to a watchglass and dry in the oven for 30 minutes. Determine the mass ($\pm$0.01 g) of the crystals and show them to the laboratory instructor. Calculate the percent yield of your alum crystals.
5. Dispose of the filtrate in the "**Waste Salts**" container.

Part E: Determine the Melting Point of the Crystalline Alum

1. Place your finely ground, dry alum to a depth of about 0.5 cm in the bottom of a melting point capillary tube.
2. In a second tube, place finely ground "control" potassium alum (Your instructor will show you how to get the alum into the capillary tubes.)
3. Use a rubber band to attach the capillary tubes to a thermometer, with the bottom of the capillary tubes beside the thermometer bulb. Support the thermometer and capillary tube (using a thermometer clamp or burette clamp) in a beaker of water such that the thermometer bulb is 0.5 cm or more above the bottom of the beaker.
4. Slowly heat the water (about 3 deg/minute), using a hot plate. Carefully watch the solid. When the solid melts, note the temperature. You can add cold water to the beaker to bring down the temperature and cause the alum to solidify (or raise the thermometer out of the water).
5. Repeat the heating cycle several times to verify your melting point. Compare the melting point of your compound to a known good sample of potassium alum. It has a melting point of 92.5°C.

What if the observed melting point of the potassium alum is "way off"? As you know the melting point of the pure alum, you can use the difference in melting point to define the melting point of your sample. Or you can calibrate your thermometer using water. Water boils at 99°C at Skyline College (about 735 Torr) and melts at 0°C. Record the thermometer temperature when water boils and when the thermometer is placed in an ice water bath. Do a slope calculation to correct your observed temperature.

What if the solid does not melt at 100 °C? It is possible that there was not enough sulfuric acid added and the solid is or contains aluminum hydroxide (check in the CRC to find it's melting point). If it is aluminum hydroxide is present, it should have been filtered out in part C.
Dispose of the capillary tube in the broken glass waste container and the alum in the labeled container.

Data Analysis:

Use stoichiometry to calculate the theoretical yield of potassium alum. Calculate your experimental yield based on your yield vs the theoretical yield.

The melting point is depressed, depending on the amount of impurities in the solid. The equation is $\Delta T^{o}_{fp} = [K_{fp}] \times \mathbf{m}$, where **m** is the molality, or moles solute per kg of solvent. If your sample has a significantly lower melting point than the control, your purity is poor.

Error Analysis Questions for Alum Synthesis Experiment

1. After collecting the alum product from the synthetic experiment, a chemist calculates a percent yield of 108.9%. What does this suggest regarding the purity of the resulting product? List two possible reasons for such a high percent yield.

2. Assuming the sulfuric acid (H_2SO_4) added is insufficient to fully react with all of the $Al(OH)_4^-$, how would this affect the theoretical yield of alum. Discuss your reasoning in the context of limiting reagents.

Synthesis of an Alum • Assignment & Report Guidelines

<u>READING</u> Experiment – Lab Manual Pages

Laboratory Handbook:
Section VII – *Separations*

- Suction Filtration – Using a Büchner Funnel

Chemistry, 6th ed. by Silberberg: Sections 3.4-3.5

<u>PRE-LAB</u> Begin the prelab on a new page of your laboratory notebook. **ALL elements of the pre-lab MUST be completed before an experiment is started.** The COPY page from your notebook will be collected as you enter the lab. The original pages must stay in your notebook.

Heading

- Title of experiment, your name, the date.

Purpose

- Briefly, but specifically explain the purpose of the experiment.

General Strategy

- **Summarize and explain** the procedure of the experiment.

Balanced Equation

- Provide the net ionic equation for the crystallization (final step) of the alum we are synthesizing. This equation is provided in the lab manual.

Data Table

- On a NEW page, prepare a table recording each quantity you will measure in the lab. (Do ***not*** turn in with the prelab.)

Answers to Pre-Lab Questions

- Answer the questions on the PRE-LAB Questions Handout. For numbers 2 & 3 – *keep the answers simple*!

LAB REPORT *Begin the lab report section on a new page of the lab notebook.* ***The discussion and conclusion sections should be word-processed.*** Other parts of the report - calculations, etc. may be typed or written in the lab notebook.

Heading

- Title of experiment, your name, the dates of the experiment.

Data / Observations / Results

- ORIGINAL QUALITATIVE & QUANTITATIVE DATA (signed data pages from your lab notebook).

Calculations / Results

Show calculations for the following quantities. Use proper format for labeling and showing calculations in a formal report):

- Stoichiometric calculation for the theoretical yield (in grams) of potassium aluminum sulfate dodecahydrate.
- Percent yield calculation.

Discussion – Theory / Results / Error Analysis

- Briefly summarize the experiment and your results.
- Discuss your percent yield and in what ways material may have been lost. What is likely the greatest source of loss? How could you reduce loss if you were to repeat the experiment? (Focus on the main source of loss.)
- Discuss the melting point, compare to the accepted value, and comment on the purity of your crystals.

Conclusions

- Comment on whether you successfully synthesized the alum you wished to make and report the percent yield and melting point of your alum.

Answers to Post-Lab Questions

- Please answer the post-lab questions on the following page.

Alum Experiment

PRE-LAB QUESTIONS Name: ________________________

1. Consider the ***overall*** reaction for the synthesis of alum below. If you start with 0.518 g of aluminum foil, calculate the mass of potassium aluminum sulfate dodecahydrate that can theoretically be produced.

$$2\ KOH_{(aq)} + 2\ Al_{(s)} + 4\ H_2SO_4 + 10\ H_2O \rightarrow 2\ KAl(SO_4)_2{\cdot}12H_2O_{(s)} + 3\ H_{2(aq)}$$

2. Your sample melted at a lower temperature than the control. Does this mean that your sample is purer or more contaminated than the control?

3. Your sample did not melt when the capillary tube containing it was placed in the boiling water. What might have happened?

Alum Experiment

POST-LAB QUESTIONS Name: ____

1. Consider one of the steps in the reaction process of the experiment we carried out:

$$2\ Al_{(s)} + 2\ KOH_{(aq)} + 6\ H_2O_{(l)} \rightarrow 2\ K^{+}_{(aq)} + 2\ Al(OH)_4^{-}{}_{(aq)} + 3\ H_{2(g)}$$

If 10.0 g of hydrogen gas were formed in the reaction, what mass of aluminum would have had to react?

2. Consider the ***overall*** reaction for the synthesis of potassium alum:

$$2\ KOH_{(aq)} + 2\ Al_{(s)} + 4\ H_2SO_4 + 10\ H_2O \rightarrow 2\ KAl(SO_4)_2{\cdot}12H_2O_{(s)} + 3\ H_{2(aq)}$$

If 15.0 g of aluminum were reacted under the proper conditions and a 72.0 % yield were achieved, what mass of alum would have been formed?