Lab AL: Preparation of Alum

Learning Objectives

To combine many of the technical skills you have gained during the semester to grow a sample of crystals.

To apply your knowledge of limiting reactants and stoichiometry to calculate a theoretical yield.

To determine a percent yield of synthesized product.

Background Information

Alums are a class of compounds with the general formula $M^{+}M^{3+}(SO_4)_2 \cdot 12\,H_2O$. The metal ion M^{+} is commonly Na^{+}, K^{+}, Tl^{+}, $NH_4{}^{+}$, or Ag^{+}, while M^{3+} is Al^{3+}, Fe^{3+}, Cr^{3+}, Ti^{3+}, or Co^{3+}.

Alums have a variety of uses in our everyday lives. Baking powder, for example, is a mixture of sodium aluminum alum ($NaAl(SO_4)_2 \cdot 12\,H_2O$) and sodium carbonate ($Na_2CO_3$). When water is added to baking powder, the aluminum ion reacts with H_2O to form H^{+}, which in turn combines with $HCO_3{}^{-}$ to form carbonic acid (H_2CO_3). Recall that carbonic acid is unstable and immediately decomposes releasing CO_2 gas. The gas released causes the cake "rise"! Ammonium aluminum sulfate ($NH_4Al(SO_4)_2 \cdot 12\,H_2O$) is used in pickling cucumbers, whereas chrome alum ($KCr(SO_4)_2 \cdot 12\,H_2O$) is used in tanning leather.

Potassium aluminum alum ($KAl(SO_4)_2 \cdot 12\,H_2O$), commonly known as "potassium alum" or "potash alum", is widely used as a coagulant in the purification of drinking water. Ancient Egyptians used potassium alum to reduce the cloudiness of water by 1500 BC!

You will be synthesizing potassium aluminum alum ($KAl(SO_4)_2 \cdot 12\,H_2O$) starting with aluminum metal (either aluminum foil or a piece of an aluminum can). Aluminum reacts rapidly with a hot aqueous solution of potassium hydroxide, KOH, to produce a soluble potassium aluminate salt (Reaction AL.1).

$$2\,Al(s) + 2\,K^{+}(aq) + 2\,OH^{-}(aq) + 10\,H_2O(l) \longrightarrow 2\,K^{+}(aq) + 2\,[Al(H_2O)_2(OH)_4]^{-}(aq) + 3\,H_2(g) \quad \text{(Reaction AL.1)}$$

When this salt is reacted with sulfuric acid (), aluminum hydroxide ($Al(H_2O)_3(OH)_3$) precipitates as shown in Reaction AL.2

$$2\,K^{+}(aq) + 2\,[Al(H_2O)_2(OH)_4]^{-}(aq) + 2\,H^{+}(aq) + SO_4{}^{2-}(aq) \longrightarrow 2\,K^{+}(aq) + SO_4{}^{2-}(aq) + 2\,Al(H_2O)_3(OH)_3(s)$$

(Reaction AL.2)

This precipitate then dissolves as additional acid is added Reaction AL.3:

$$2\,Al(H_2O)_3(OH)_3(s) + 6\,H^{+}(aq) + 3\,SO_4{}^{2-}(aq) \longrightarrow 2\,Al^{3+}(aq) + 3\,SO_4{}^{2-}(aq) + 12\,H_2O\,(l) \quad \text{(Reaction AL.3)}$$

Since the resulting solution contains K^+, Al^{3+}, and $SO_4{}^{2-}$ ions, potassium aluminum sulfate precipitates as shown in Reaction AL.4 as octahedrally shaped crystals when a nearly saturated solution is cooled to 0 °C.

$$K^{+}(aq) + Al^{3+}(aq) + 2\,SO_4{}^{2-}(aq) \longrightarrow KAl(SO_4)_2 \cdot 12\,H_2O\,(s) \quad \text{(Reaction AL.4)}$$

As part of your Lab Report, you'll be asked to write a complete balanced chemical equation for the conversion of the starting material, Al(s), to the product, $KAl(SO_4)_2 \cdot 12\,H_2O(s)$. The Procedure Video on LabFlow describes how to combine Equations AL1-4 to come up with this complete balanced equation, just skip to the 29:00 timestamp in the video.

Experimental Procedure

Safety Precautions

Wear safety glasses and a lab coat/apron when performing this experiment.

1. Obtain two small pieces of aluminum foil from the reagent shelf. Using a top-loading balance, weigh the two pieces of aluminum foil together, and record the actual mass in Report Table AL.2. (The total mass should be around 1 g.)

2. Add the metal to a 250-mL or larger beaker. Place the beaker on a hot plate, positioned under the mushroom hood on the benchtop.

3. Carefully add 25 mL of 3.0 M potassium hydroxide, KOH, solution to the beaker containing the aluminum.

CAUTION

KOH is exceedingly caustic. Do not spatter the resulting mixture. If any spills or spatters occur onto your skin or clothing, rinse the affected area thoroughly with water.

4. Begin heating the beaker gently on the hot plate, set to a temperature around 100 °C. Performing the heating under the hood allows the hydrogen gas that is evolved to be pulled out of the laboratory. Continue heating with frequent stirring until all of the aluminum has reacted. If necessary, carefully add distilled water to the beaker to maintain nearly 25 mL of solution. Ensure that all of the aluminum reacts, as your yield of alum depends greatly on this step.

5. After all of the aluminum has reacted, add 25 mL of distilled water. Prepare a gravity filtration apparatus with a plastic funnel set into a 150-mL or larger beaker, (**Performing a Gravity Filtration Video**). Filter the warm solution through the filter paper.

6. Permit the resulting clear solution, which contains K^+, $[Al(H_2O)_2(OH)_4{}^-]$, and excess OH^-, to cool to room temperature.

7. Slowly acidify the solution by adding small amounts of 6.0 M sulfuric acid (H_2SO_4) solution until a total of

30.0 mL has been added. As you add the H_2SO_4, a precipitate first forms, and then redissolves as more acid is added. If all of the precipitate does not redissolve after the addition of 30.0 mL of acid is complete, heat the mixture gently. You must have a clear, colorless solution. Continue to stir, heat the solution to boiling, and reduce the volume of solution to 50. mL

8. Cool the solution in an ice bath for 25-30 minutes without any agitation. If the solution is maintained as motionless as possible, well-formed octahedral crystals of alum should grow in the beaker. If no crystals form, reheat the solution to evaporate an additional 10. mL of water and re-cool the solution in the ice bath.

9. Weigh a clean, dry watch glass together with a piece of filter paper on a top-loading balance. Record the combined mass in Report Table AL.2.

10. After all of the solid alum has formed, filter the mixture using vacuum filtration, and wash the resulting crystals with 15 mL of a 50:50 (by volume) water-alcohol solution. Allow the vacuum to pull on the sample for about 2 minutes.

11. Transfer the filter paper and crystals to the watch glass and place them in the laboratory oven for at least 30 minutes.

12. Carefully remove the watch glass with a pair of tongs, and set it on a piece of iron gauze to cool. Once cool, weigh the dry crystals and the watch glass on a top-loading balance and record the mass in Report Table AL.2. Your instructor will tell you what to do with your alum sample. You may be asked to add it to a storage container for students in another course to analyze for the sulfate content.

Name: ____________________

Section: __________ Date: __________

Data

Report Table AL.1: Mass Data for Preparation of Alum

Mass of aluminium foil (g)	______________
Mass of filter paper + watch glass (g)	______________
Mass of watch glass + dry alum (g)	______________

Instructor's Initials: __________

Calculations

Report Table AL.2: Mass Data for Preparation of Alum

Mass of dry alum (g)	______________
Theoretical yield (g)	______________
Percent yield (%)	______________

Sample Calculations

Balanced chemical equation for conversion of Al(s) to ($KAl(SO_4)_2 \cdot 12\,H_2O$) in aqueous solution:

Theoretical Yield:

Percent Yield:

Questions to Consider

1. What is the precipitate which forms and then redissolves upon adding H_2SO_4 to the mixture of K^+, $[Al(H_2O)_2(OH)_4^-]$ and OH^-?

2. The percent yield of alum is calculated based on the starting quantity of aluminum. Why can't the quantity of sulfuric acid be used to calculate the percent yield? Indicate clearly your calculations and reasoning.

3. Is your experimental yield of alum greater than, less than or equal to the theoretical yield? Give specific reasons as to why this might the case.

4. If crystals do not form upon cooling the solution of alum, why is an additional 10. mL of water evaporated from the solution?