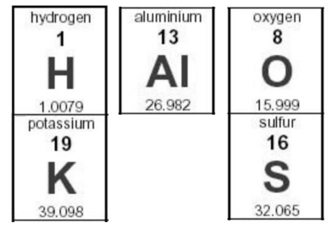
Experiment: Synthesis of Alum

Minneapolis Community and Technical College

v. 10.17

Objectives: To synthesize alum from aluminum foil, predict the theoretical product yield, measure the actual yield and calculate a percent yield.

Prelab Questions: Read through this lab handout and answer the following questions before coming to lab. There will be a quiz at the beginning of lab over this handout and its contents.



1. What is the molar mass of Alum? $AlK(SO_4)_2 \cdot 12H_2O$
2. What is a hydrate?
3. How many aluminum atoms are required to make one unit of alum?
4. Where does the sulfate ion in the alum product come from?
5. Where does the potassium ion in the alum product come from?
6. What's the difference between gravity and vacuum filtration?
7. How should the end of the funnel be positioned when performing a gravity filtration?
8. What is the purpose of the glass stirring rod when decanting?
9. What is the correct way to construct an ice bath?
10. When do alum crystals form in the experiment?
11. How many grams of Alum can be theoretically formed if there were 0.3506 grams of aluminum initially?

Alum is a material that has many uses and is commercially available in the baking isle of most grocery stores. Traditionally, cooks have used alum as part of the pickling process where it works both to preserve and firm up the pickles. In water purification processes, dissolved alum reacts with negatively charged particles producing a gelatinous precipitate (a.k.a. a "floc") that can be removed from the water sample after settling.

In this activity, you will be preparing potassium alum from aluminum metal using sulfuric acid and potassium hydroxide. Alum's chemical formula, **$AlK(SO_4)_2 \bullet 12H_2O$** tells us that not only is alum a salt (metal/non-metal) but it's also a hydrate with twelve water molecules imbedded in the salt's crystalline structure.

Thus, the chemical name for alum is **aluminum potassium sulfate dodekahydrate**.

A series of chemical reactions occurs as you convert aluminum metal into alum. The first dissolves the aluminum metal when immersed in hot, aqueous potassium hydroxide. The reaction also liberates hydrogen gas.

$$2Al_{(s)} + 2KOH_{(aq)} + 6H_20_{(l)} \rightarrow 2K^+_{(aq)} + 2Al(OH)_4^-{}_{(aq)} + 3H_{2(g)}$$

Next, sulfuric acid is added to the reaction mixture producing an aluminum hydroxide solid precipitate.

$$2Al(OH)_4^-{}_{(aq)} + H_2SO_{4(aq)} \rightarrow 2Al(OH)_{3(s)} + 2H_2O_{(l)} + SO_4^{2-}{}_{(aq)}$$

With additional sulfuric acid and continued stirring, the aluminum hydroxide precipitate re-dissolves:

$$2Al(OH)_4^-{}_{(aq)} + 3H_2SO_{4(aq)} \rightarrow 2Al^{3+}_{(aq)} + 3SO_4^{2-}{}_{(aq)} + 6H_2O_{(l)}$$

When the products of this last reaction are cooled in an ice bath, the final alum product precipitates:

$$K^+_{(aq)} + Al^{3+}_{(aq)} + 2SO_4^{2-}{}_{(aq)} + 12H_2O_{(l)} \rightarrow KAl(SO_4)_2 \cdot 12H_2O_{(s)}$$

This last reaction is essentially the net reaction that gives us the mole ratio connecting alum with its key reactants.

Experiment: Aluminum/KOH reaction

1. Using an analytical balance, pre-weigh a 250 mL beaker.

2. Use a scissors to cut up your aluminum foil and place between 0.9 g and 1.1 grams of aluminum foil pieces the beaker.

3. Reweigh the beaker and aluminum foil to determine the mass of the aluminum to the nearest 0.0001 g.

4. While working in the hood, use the provided graduated cylinder to *carefully* add approximately 50 mL of 1.4 *M* KOH (potassium hydroxide) to the reaction beaker containing the aluminum foil.

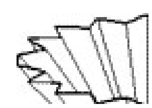
5. Continue working in the hood and warm the reaction mixture using a hotplate. ($200^{o}C$).

6. Stir the contents of the beaker with a glass stirring rod to keep the aluminum foil in solution as it will sometimes stick to the sides of the beaker.

7. When the reaction is complete (you decide) remove the beaker from the hotplate

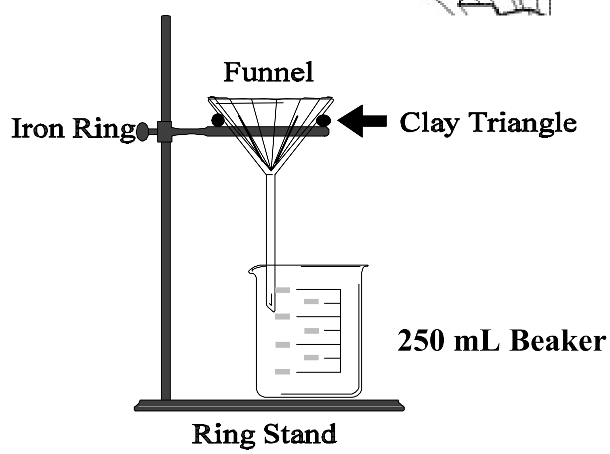
8. Remove the beaker from the hotplate and allow it to cool in the hood for several minutes.

Experiment: Gravity Filtration



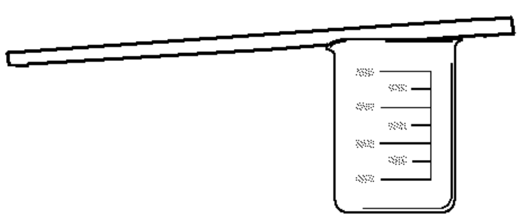
1. Assemble the gravity filtration apparatus (figure at right) in your hood space.

   Your instructor will demonstrate how to flute (fold) your filter paper for this step. (right)



2. When cool enough to handle, pour the contents of the 250 mL reaction beaker into the gravity filtration apparatus assembled above.

   Use a glass stirring rod (right) to guide the liquid into the funnel. Don't overfill the funnel.



3. Dispose of the gravity filter paper as instructed (your instructor may pick it up for you).

4. Clean, rinse and dry the filter funnel before returning it to the ring stand.

Experiment: Alum Production and Crystallization

1. Measure the temperature of your reaction mixture using a glass thermometer and record this on your data sheet.

2. Continue working in the fume hood as you **slowly pour** approximately 20 mL of 9 M H_2SO_4 into the beaker containing the filtered liquid while carefully stirring with the thermometer.

3. Record the final temperature reached on your data sheet.

4. Construct an ice bath from a 600 mL beaker by first filling it ¾ full with ice and then adding small amounts of TAP water (not distilled) to cover the ice. A properly constructed ice bath should be mostly ice with just enough water to improve contact with the beaker that's being cooled speeding up the cooling process.

5. Place the 250 mL beaker in the ice bath and use a glass stirring rod to *vigorously* stir the mixture.

   Rub/scrape the sides of the beaker with a stirring rod to accelerate the formation of product crystals.

6. Cool for at least 10 minutes and when no further changes are observed measure the mixture's temperature and record it as the "Recrystallization temperature" with one decimal place accuracy.

Experiment: Vacuum Filtration of Alum Product

1. Obtain a piece of vacuum filter paper.

2. Weigh the watch glass and vacuum filter paper on the analytical balance. Record the masses in your data table.

3. The vacuum filtration apparatus will be set up for you in the lab. Check to make sure the filter flask is secure in the clamp before proceeding.

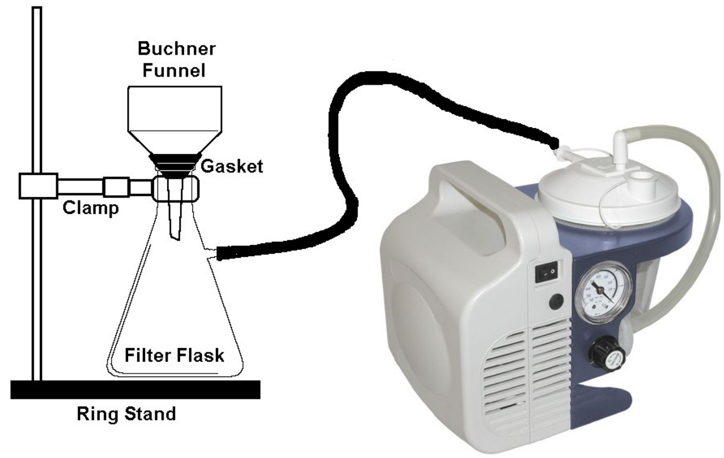
4. Place the pre-weighed filter paper in the Buchner funnel.

5. Use a small amount of water to wet the filter paper. This will "stick" the filter paper and keep it in place during the initial filtering stages.

6. Turn on the vacuum pump.

7. Remove the 250 mL beaker from the ice bath and swirl the contents to suspend the solid alum crystals in the liquid.

8. Quickly pour the 250 mL beaker's contents in to the Buchner funnel. (Don't spill)



9. Rinse any remaining alum from the beaker into the Buchner funnel using ice cold distilled water.

10. Continue filtering while you wash the alum product with small amounts of ice cold distilled water. You may turn off the vacuum pump briefly to let the alum crystals momentarily bathe in the cold water before re-applying suction.

11. Continue running the vacuum pump for 2 minutes to remove as much liquid as possible.

12. Carefully remove the Buchner funnel from the Filter Flask. Use a metal spatula to transfer the alum and filter paper from the funnel onto the pre-weighed watch glass.

13. Write your name and lab section number on a small piece of paper.

14. Place your watch glass and alum on the tray provided by your instructor. Slip the name tag you just created between the filter paper and watch glass. This will let you easily identify your alum from others when you return to lab later.

15. You will weigh your watch glass, filter paper, and **dry alum** when you return to lab. At that time you'll be able to finish your calculations.

Experiment: Acid Neutralization Procedure and Disposal

1. Empty the filter flask into a 100 mL graduated cylinder.
   Record the volume of this liquid (called the "filtrate") on your data sheet.

2. Transfer the liquid from the graduated cylinder to the empty 600 mL beaker.

3. Place the beaker in the bottom of a sink and neutralize the contents by adding scoops of solid sodium bicarbonate ($NaHCO_3$) to the solution while stirring with a glass stirring rod.

4. When additional $NaHCO_3$ no longer produces effervescence (fizzing), you can flush the beaker's contents down the drain with tap water.

Experiment: Cleanup

1. All glassware should be thoroughly washed and then rinsed with a small amount of distilled water. Leave the clean glassware on a dry paper towel at your bench top.

2. Clean the Buchner funnel and filter flask by rinsing them carefully with tap water followed by a small amount of distilled water.

3. Place the Buchner funnels upside down near each filter station.

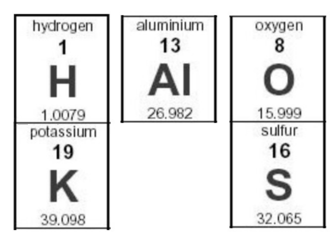
4. Wipe down your laboratory spaces (benchtop and hood) with a damp sponge.

IMPORTANT

Your experimental report is due when you return to lab next week. However, you won't be able to finish your calculations until you have weighed your dried alum product.

Therefore, you should complete as much of the report as possible before returning to lab (questions and calculations). Once you have your final dried alum mass, the remaining calculations will take only a few minutes to complete.

Prelab Questions: Answers



1. 474.3796 g/mol
2. "In chemistry, a hydrate is a substance that contains water or its constituent elements." (https://en.wikipedia.org/wiki/Hydrate)
3. 1 aluminum atom is required to form 1 alum formula unit.
4. H_2SO_4
5. KOH
6. Gravity filtration utilizes the weight of the solution to push it through filter paper. Vacuum filtration is also aided by gravity, but suction mainly pulls the solution through the filter paper.
7. In the beaker touching the lower beaker's inside wall surface (Reduces splashing).
8. The stirring rod directs (aims) the liquid being poured accurately.
9. Mostly ice. Just enough water to fill in the voids.
10. When the final solution is cooled. Alum is less soluble at cold temperatures and so by cooling the solution solid alum will form.
11. 6.16 grams of alum can theoretically form.

C1151 Data Sheet
Synthesis of Alum
Instructor Initials_____

Name_________________________
Date of Exp. ________________
Lab Section ________________

Synthesis of Alum

Data Table	Measurements (units)
250 mL Beaker mass	
250 mL Beaker + aluminum mass	
Temperature before & after adding H_2SO_4	Before / After /
Recrystallization temperature	
Recovered filtrate volume	
Filter paper mass	
Watch glass mass	
Final filter paper, watch glass & dry alum mass	
Alum mass obtained	
Aluminum mass	
Aluminum moles	Excess Significant Figures
Theoretical alum moles	Excess Significant Figures
Molar Mass for $AlK(SO_4)_2 \bullet 12H_2O$	Excess Significant Figures
Theoretical alum mass	Excess Significant Figures
Alum percent yield (Before filtrate adjustment) **(See question 2)**	CORRECT Significant Figures
Alum percent yield (After filtrate adjustment) **(See questions 3 & 4)**	CORRECT Significant Figures

Questions: (Continued on next page)

1. Is the reaction involving the addition of sulfuric acid an exothermic or endothermic reaction? Why?

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2. Determine the theoretical alum yield in the space below. Neatly show all work and record this result in the data table.

3. Determine the percent yield for alum in the space below. Neatly show all work and record this result in the data table.

4. Although most of the alum you produced was recovered on the filter paper, some amount of alum *remained dissolved*, passed through the filter paper and is now un-weighable. However, we can determine how much is dissolved as follows:

   a. The amount of alum that can be dissolved in water to make a 50 mL solution is known
   1.4 g of alum @ 1.0°C **1.9 g of alum @ 3.0°C** **2.4g of alum @ 6.0°C**

   Using MS Excel, construct a mass vs. temperature scatter graph using these three points. Perform a trendline analysis and ***attach the graph to your report.***

   b. Use the trendline analysis result and the temperature you recorded earlier to determine how much alum should still be dissolved in 50 mL of solution at this temperature. Neatly show your work below.

   c. Use your result from the previous step (b) to determine the amount of alum you predict is dissolved in your filtrate volume. Neatly show your work below.

   d. Add the dissolved alum mass to the actual product weight and use this value to recalculate the percent yield (Show calculations). Report this value on the data table indicated by (After filtrate adjustment)

5. How many grams of aluminum were used to prepare 15.5 g of alum if the percent yield was known to be 78.2%? Show your work neatly on the back of this page.