AP Chemistry Lab #2- Synthesis and Analysis of Alum (Big Idea 1 & 2)

Ask a Question: How can I utilize chemical synthesis to produce potassium aluminum sulfate dodecahydrate and to quantify the identity and purity of this substance using several analytical tests?

The term *alum* is a general family name for a crystalline substance composed of cations with 1+ and 3+ charges. Alum is used in water treatment facilities because when it is added to water alum reacts with the bicarbonate alkalinities present in water and forms a gelatinous precipitate. This loosely packed clump of fine particles, called floc, attracts other fine particles and suspended material in water, and settles down at the bottom of the container. In this experiment, you will synthesize a type of alum called potassium aluminum sulfate dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$. You will synthesize this compound by placing the appropriate ions in one container in aqueous solution and then evaporate the water to form the alum crystals.

This particular compound has been chosen because it is relatively simple to prepare a pure sample. The process of synthesizing this compound is interesting in that it involves both chemical and physical reactions. Chemically, aluminum is oxidized from aluminum foil to prepare the Al^{3+} ions. Physically, as the solution that contains the mixture of ions evaporates, crystals will form which contain six waters of hydration bonded to the aluminum ion and six waters bonded to the potassium ion.

Aluminum is considered a reactive metal, but because its surface is usually protected by a thin film of aluminum oxide, it reacts slowly with acids. It does, however, dissolve quickly in basic solutions. Excess hydroxide ion converts the aluminum to the tetrahydroxoaluminate ($Al(OH)_3$) precipitates. Continued addition of acid causes the hydroxide ions to be completely neutralized, and the aluminum exists in solution as the hydrated ion $[Al(H_2O)_6]^{3+}$. Aluminum hydroxide is considered to be an amphoteric hydroxide because it dissolves in both acids and bases.

After a compound has been synthesized, tests should be carried out to verify that the compound formed is indeed the compound desired. There are a number of tests that can be performed to verify that the compound is the one desired. The first test is to find the melting temperature of the compound and compare this value with the accepted (published) value for alum (92.5°C). The second test determines the water of hydration present in the alum crystals. The third test is a chemical test to determine the percent sulfate in your sample of alum.

Part 1 Materials:

250 mL beaker	aluminum foil	fume hood
25 mL or 50 mL graduated cylinder	3 M sulfuric acid solution, H_2SO_4	hot plate
Büchner funnel and filter flask	baking soda, $NaHCO_3$	balance
watch glass	3 M potassium hydroxide solution, KOH	
glass stirring rod	aqueous ethanol solution, 50%	
lab burner, ring stand, ring, wire gauze	vinegar, dilute CH_3COOH solution	
plastic wrap or Parafilm®	ice bath	

Part 1 Procedure:

1. Obtain and wear goggles.

2. Obtain a piece of aluminum foil and measure its mass. For best results, you should have about 1.00 g of aluminum. Tear the foil into small pieces and place the pieces in a 250 mL beaker.

3. Set up a Büchner funnel and filter flask so that you are ready to filter the reaction mixture that will be produced in Step 4.

4. Conduct the first part of the synthesis. CAUTION: Potassium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.

Use a graduated cylinder to measure out 25 mL of 3 M KOH solution.

Slowly add the KOH solution to the beaker of aluminum pieces. Notice that the reaction is exothermic. Allow the reaction to proceed until all of the foil is dissolved.

Carefully pour the reaction mixture through your Büchner funnel and filter flask setup, and rinse the filter paper with a small amount of distilled water. Note: The reaction mixture contains three ions: K^+, $[Al(OH)_4^-]$, and excess OH^-.

Rinse the beaker with distilled water, and pour the filtered liquid back into the beaker.

5. Allow the solution to cool to near room temperature. If you are pressed for time, you may cover the beaker with plastic wrap or Parafilm, and store the liquid overnight.

6. Clean the Büchner funnel and filter flask, and prepare it for more filtering that you may need to do in Step 7/10.

7. Complete the synthesis.

Use a graduated cylinder to measure out 35 mL of 3 M H_2SO_4 solution. CAUTION: The reaction mixture must be cooled to room temperature before proceeding. Handle the H_2SO_4 solution with care. It can cause painful burns if it comes in contact with the skin.

After the reaction mixture has cooled, slowly add the sulfuric acid solution to the beaker of liquid. Stir the mixture constantly. The reaction is strongly exothermic, so be careful as you stir the mixture. Note that aluminum hydroxide will precipitate initially. It will dissolve as more sulfuric acid is added.

If there is some solid remaining in the beaker after the 35 mL of sulfuric acid has been added, pour the mixture through the Büchner funnel and filter flask to separate the undissolved solid from the mixture.

8. Gently boil your mixture until you have about 50 mL of liquid in the beaker.

9. Cool the beaker of solution. Choose one of the two methods listed below.

Allow the solution to cool overnight. In most cases, this gradual cooling forms a good crop of alum crystals.

Prepare an ice bath for the 250 mL beaker. Place your beaker of solution, uncovered, in the ice bath. Do not move the ice bath or the beaker. After about fifteen minutes, crystals of alum will appear in the beaker. If there are no crystals after fifteen minutes, scrape the bottom of the beaker with a glass stirring rod to create a rough spot for crystal growth. You may also heat the solution to evaporate more water and cool the solution again.

10. Collect your alum crystals by pouring them onto the Büchner funnel and filter-flask setup. Use vacuum filtration to wash the crystals on the filter paper with 50 mL of an aqueous ethanol solution (50%). The crystals will not dissolve in this solution.

11. Remove the filter and crystals from the Büchner funnel and allow the crystals to dry at room temperature. Measure and record the mass of your sample of alum. Store the crystals for further analysis.

Part 1: Research the question and collect Data and Evidence:

Q1. Determine the theoretical yield of the alum. Use the aluminum foil as the limiting reagent and presume that the foil was pure aluminum.

Q2. Calculate the percent yield of your alum crystals.

Q3. Discuss the factors that affected the percent yield.

Q4. Write the balanced net ionic equations for the following: (a) aluminum and potassium hydroxide, yielding $[Al(OH)_4]^-$ and hydrogen gas; (b) hydrogen ions and $[Al(OH)_4]^-$, yielding aluminum hydroxide; (c) aluminum hydroxide and hydrogen ions, yielding $[Al(H_2O)_6]^{3+}$; and (d) the formation of alum from potassium ions, sulfate ions, $[Al(H_2O)_6]^{3+}$, and water.

Q5. Describe a synthesis reaction.

Q6. Describe how the solubility of alum in various solvents and water at different temperatures was used in conducting the experiment.

Q7. Write a detailed description of the alum crystals or include a picture.

Part 2 Materials:

Test #1

LabQuest
LabQuest App
Temperature Probe
capillary tubes
150 mL beaker or Thiele melting-point tube
mortar and pestle
glass stirring rod
alum crystals, $KAl(SO_4)_2 \cdot 12H_2O$
hot plate
ring stand, ring, and wire gauze
utility clamp
cork or split stopper
small rubber band

Test #2

Alum crystals, $KAl(SO_4)_2 \cdot 12H_2O$
ring stand, ring, and clay triangle
tongs or forceps
crucible with cover
balance
lab burner

Test #3

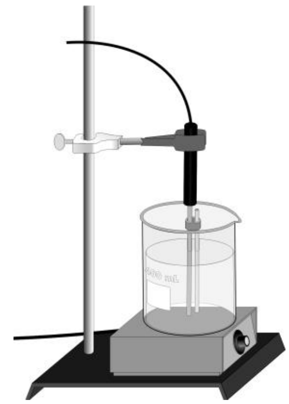
0.20 M barium nitrate, $Ba(NO_3)_2$, solution
watch glass to fit the 250 mL beaker
50 mL or 100 mL graduated cylinders
Gooch crucible filter apparatus or filter paper
rubber policeman or other scraping tool
Alum crystals, $KAl(SO_4)_2 \cdot 12H_2O$
ring stand, ring, and wire gauze
glass stirring rod
250 mL beaker
balance

Part 2 Procedures:

Test #1: Determine the Melting Temperature

1. Obtain and wear goggles.
2. Connect the Temperature Probe to LabQuest and choose New from the File menu.
3. Use a mortar and pestle to pulverize about 0.5 g of dry alum and place it in a small pile in the mortar. Push the open end of a capillary tube into the pile of the alum powder. Pack alum into the capillary tube to a depth of about 1 cm by tapping the tube lightly on the tabletop.

4. Use a rubber band to fasten the capillary tube to the Temperature Probe. The tip of the tube should be even with the tip of the probe. Use a utility clamp to connect the Temperature Probe to a ring stand. If necessary, place the probe in a split stopper or a cork to secure it in the clamp (see figure to the right).



5. Prepare a water bath to be heated by a hot plate. Your instructor may also direct you to use a Thiele tube. If you do not use a Thiele tube, stir the water bath throughout the testing to maintain a constant bath temperature.

6. Monitor the temperature readings on the Main screen. Immerse the capillary tube and Temperature Probe in the water bath. Warm the alum sample at a gradual rate so that you can precisely determine the melting temperature. The white powder will become clear when it is melting. Observe the temperature readings and record the precise melting temperature when the substance is completely clear.

7. Conduct a second test with a new sample of alum in a new capillary tube.

Test #2: Determine the Water of Hydration

8. Heat a crucible with cover over a burner flame until it is red hot. Allow the crucible to cool, and then measure the total mass of crucible and cover. Handle the crucible with tongs or forceps to avoid getting fingerprints on it.

9. Place about 2 g of your alum crystals in the crucible, and then measure the mass of the crucible, cover, and alum. Record this measurement in the data table.

10. Select one of the two drying methods listed below.

a). Set up a ring, ring stand, and triangle over a lab burner. Use tongs or forceps to set the crucible at an angle on the triangle and place the cover loosely on the crucible. Use a lab burner to *very gently* heat the crucible of alum until you can see no vapor escaping from the crucible. It is important that the vapor does not carry any alum with it. After the vapor is gone, heat the crucible more strongly for five minutes, and then cool the crucible. b). Place the crucible in an oven overnight at 110°C.

11. Measure and record the mass of crucible, cover, and alum after drying.

12. Reheat the crucible and alum sample for five additional minutes. Cool and measure the mass of the crucible again. If the two masses are the same (or very nearly so), the test is done. If not, repeat the heating and weighing until a constant mass is obtained.

Test#3: Determining the Percent Sulfate

13. Obtain a clean, dry Gooch crucible and measure its mass. If you must clean the crucible, follow the procedure in Step 14. If not, proceed directly to Step 15. If you are not using a Gooch crucible, then use the finest-grain filter paper available, such as Whatman No. 42, because the very tiny particles of barium sulfate precipitate will pass through most paper filters. Measure and record the mass of the filter paper, and then proceed to Step 15.

14. Measure the mass of about one gram of your alum sample into a 250 mL beaker. Add about 50 mL of distilled water to the beaker of alum and stir the mixture to dissolve the entire solid.

15. Calculate the volume of 0.20 M $Ba(NO_3)_2$ solution that is needed to completely precipitate the sulfate ions in the beaker of alum solution. Measure out twice the volume that you have calculated, and slowly add it to the beaker of alum solution. Stir the mixture to ensure complete mixing of the reagents. *Handle the barium nitrate solution with care. This solution is toxic.*

16. Select one of the two methods below for preparing the precipitate: a). Set up a ring stand, ring and wire gauze for heating over a lab burner. Place a watch glass over the beaker and heat the beaker of your reaction mixture over a lab burner. Heat the mixture to near boiling for 15 minutes. This step helps collect the particles of precipitate to a larger size and eases the filtering process. b). Use a watch glass to cover the beaker of reaction mixture. Store the beaker in a safe place overnight.

17. Allow the mixture to cool. Filter the beaker of precipitate through the Gooch crucible with suction (or filter paper and funnel). Add liquid to the crucible very slowly, because the barium sulfate precipitate tends to creep up the sides of the container. Use a rubber policeman to scrape all of the precipitate from the beaker to the crucible. Wash the beaker and the crucible several times with small amounts of distilled water.

18. Carefully move the crucible (or filter paper) of precipitate to a small beaker and place it in a drying oven. If you are using filter paper, either air dry the paper or place it in an oven that is no warmer than 50°C to prevent charring of the filter paper. After the precipitate is dry and cool, measure and record its mass.

Data Table:

Test #1 Melting Temperature Test Results	Trial 1	Trial 2
Melting temperature (°C)		
Test #2 Water of Hydration Test Results	Trial 1	Trial 2
Mass of crucible and cover (g)		
Mass of crucible, cover, and alum before heating (g)		
Mass of crucible, cover, and alum after 1st heating (g)		
Mass of crucible, cover, and alum after 2nd heating (g)		
Mass of crucible, cover, and alum after final heating (g)		
Test #3 Percent Sulfate Test Results	Trial 1	Trial 2
Mass of Gooch filter *or* filter paper (g)		
Mass of alum before reaction (g)		
Mass of Gooch filter *or* filter paper and precipitate (g)		

19. **M**ake your Claim to address the overarching question.

20. **S**ummarize and Reflect upon the investigation. In your summary be sure to incorporate the following question:

- Is your sample alum? Use the results of the three tests to support your answer. Discuss the accuracy of your tests and possible sources of experimental error.
- Suggest other tests that could be conducted to verify the composition of your alum.
- If the melting temperature test was the only test that you conducted, how confident would you be in the identification of your sample? Explain.

Please self-assess your lab report using the STEM Journal checklist/rubric.