

The Analysis of Alum

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. In Experiment 15A, you prepared alum crystals, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$. In Experiment 15B, you will conduct a series of tests to determine if your crystals are really alum.

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.

OBJECTIVES

In this experiment, you will

- Determine the melting temperature of a sample of alum.
- Determine the water of hydration of a sample of alum.
- Determine the percent sulfate of a sample of alum.
- Verify the chemical formula of a sample of alum.

MATERIALS

Part I, Melting Temperature Test Materials

Vernier computer interface
computer
Temperature Probe
capillary tubes
150 mL beaker or Thiele melting-point tube
mortar and pestle
glass stirring rod

alum crystals, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$
hot plate
ring stand, ring, and wire gauze
utility clamp
cork or split stopper
small rubber band

Part II, Water of Hydration Test Materials

crucible with cover
balance
lab burner

alum crystals, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$
ring stand, ring, and clay triangle
tongs or forceps

Part III, Percent Sulfate Test Materials

250 mL beaker
watch glass to fit the 250 mL beaker
balance
Gooch crucible filter apparatus or filter paper
rubber policeman or other scraping tool

alum crystals, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$
0.20 M barium nitrate, $\text{Ba}(\text{NO}_3)_2$, solution
ring stand, ring, and wire gauze
50 mL or 100 mL graduated cylinders
glass stirring rod

PROCEDURE

Part I Determine the Melting Temperature of Alum

1. Obtain and wear goggles.
2. Connect a Temperature Probe to Channel 1 of the Vernier computer interface. Connect the interface to the computer with the proper cable.
3. Start the *Logger Pro* program on your computer. Open the file "15b Alum" from the *Advanced Chemistry with Vernier* folder.
4. 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 table top.
5. 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 1).

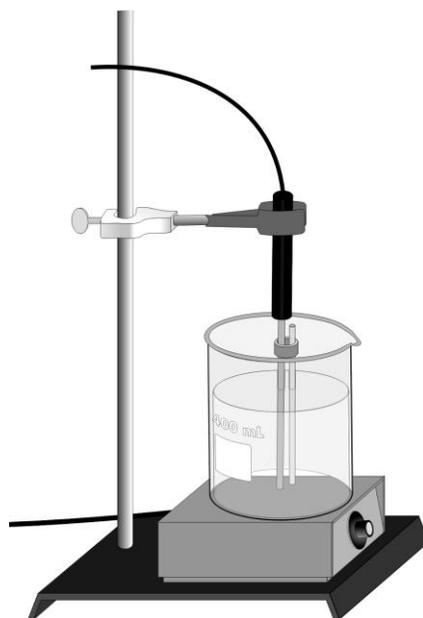


Figure 1

6. 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.
7. Click to begin data collection. 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. Data will be collected for 10 minutes. You may click to end data collection early if the substance has melted completely.

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

Part II Determine the Water of Hydration of Alum

9. 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 the crucible and cover. Handle the crucible with tongs or forceps to avoid getting fingerprints on it.
10. 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.
11. Select one of the two drying methods listed below.
 - 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.
 - Place the crucible in an oven overnight at 110°C.
12. Measure and record the mass of the crucible, cover, and alum after drying.
13. 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.

Part III Determine the Percent Sulfate of Alum

14. Obtain a clean, dry Gooch crucible and measure its mass. If you must clean the crucible, follow the procedure in Step 15. If not, proceed directly to Step 16. 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.
15. To clean a Gooch crucible, assemble the crucible, Walters adapter, and filter flask. Use suction to draw distilled water through the filter pad. Place the crucible in a beaker and dry it in an oven.
16. 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 sample.
17. Calculate the volume of 0.20 M Ba(NO₃)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. **CAUTION:** *Handle the barium nitrate solution with care. This solution is toxic.*
18. Select one of the two methods below for preparing the precipitate:
 - 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.
 - Use a watch glass to cover the beaker of reaction mixture. Store the beaker in a safe place overnight.

Computer 15B

- Allow the mixture to cool. Filter the beaker of precipitate through the Gooch crucible with suction (or use a 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.
- 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

Part I Melting Temperature Test Results	Trial 1	Trial 2
Melting Temperature ($^{\circ}\text{C}$)		

Part II 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 1 st heating (g)		
Mass of crucible, cover, and alum after 2 nd heating (g)		
Mass of crucible, cover, and alum after final heating (g)		

Part III 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)		

DATA ANALYSIS

1. 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.

2. Suggest other tests that could be conducted to verify the composition of your alum.

3. If the melting temperature test was the only test that you conducted, how confident would you be in the identification of your sample? Explain.