



P.O. Box 219 • Batavia, Illinois 60510 • 1-800-452-1261 • flinn@flinnsci.com • Visit our website at: www.flinnsci.com

Analysis of Alum, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$

AP Chemistry Laboratory #2

Catalog No. AP6354

Publication No. 6354A

Introduction

When a compound is synthesized, tests are carried out to confirm whether the compound formed is indeed the compound desired. There are a number of tests that can be performed to verify the identity of a compound. In this experiment several tests are carried out to determine if sample crystals are aluminum potassium sulfate (alum).

Concepts

- Percent composition
- Water of hydration
- Molecular formula

Background

Every compound has a unique set of chemical and physical properties. To identify a compound with certainty, a minimum number of these properties must be verified experimentally. In this experiment, three properties of a sample of aluminum potassium sulfate, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, are determined—its melting point, the number of moles of water of hydration in the formula, and percent composition of sulfate.

The first test in verifying the identity of alum is to find the melting point of the compound and compare it to the published value for alum. A small quantity of alum is powdered and placed in a capillary tube which is attached by a rubber band to a thermometer bulb. The crystals are heated in a water bath, and the temperature at which they melt is recorded and compared to reported values.

The alum is next analyzed for water of hydration. When an ionic compound is prepared in aqueous solution and isolated by crystallization, water molecules are often incorporated into the crystal structure of the compound in fixed proportions. The amount of water incorporated, referred to as the water of hydration, cannot be predicted for any compound, but must be determined experimentally. In order to determine the formula moles of water of hydration for alum, a portion of the alum will be placed in a crucible and weighed. The crucible is heated until all of the water of hydration is driven off. The crucible is then cooled and its mass measured. From the mass of the dry crystals and the mass of the water lost, the ratio of the moles of H_2O to the moles of $\text{AlK}(\text{SO}_4)_2$ is calculated and compared to the correct formula values.

The third test is a determination of the percent of sulfate in the compound. A weighed quantity of alum is dissolved in distilled water. An excess of barium ions is added to the solution to precipitate all of the sulfate as barium sulfate. The precipitated barium sulfate is filtered off, dried, and its mass determined. From the mass of the barium sulfate and the initial mass of alum, the percent sulfate is calculated and compared with the theoretical percent found from the formula.

COPY

→ Experiment Overview

The purpose of the experiment is to analyze alum, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, by three techniques in order to verify its identity. The following properties will be determined—melting point, mole ratio of hydrated water to anhydrous potassium aluminum sulfate, and percent of sulfate ion contained in the compound. Each of these properties will be compared to the literature or calculated values for alum.

NO

Pre-Lab Questions

1. When measuring a melting point, why is it necessary to raise the temperature very slowly when approaching the melting temperature?
2. Washing soda is a hydrated compound whose formula can be written $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$, where x is the number of moles of H_2O per mole of Na_2CO_3 . When a 2.123 g sample of washing soda was heated at 130°C , all of the water of hydration was lost, leaving 0.787 g of anhydrous sodium carbonate. Calculate the value of x .
3. The formula for epsom salts is $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. If 1.250 g of the compound is dissolved in water, calculate the number of milliliters of 0.200 M $\text{Ba}(\text{NO}_3)_2$ that would be required to precipitate all of the sulfate ions as barium sulfate.

Make the same determination for 1.000 g of alum.

COPY

→ Materials

Part 1

Aluminum potassium sulfate, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, 0.5 g	Mortar and pestle
Beaker, 150-mL (or Thiele melting point tube)	Ring stand, ring, and wire gauze
Bunsen burner	Rubber band
Hot plate	Thermometer, 0°C – 100°C
Capillary tube	Universal clamp
Split stopper to hold thermometer	

Part 2

Aluminum potassium sulfate, 2 g	Ring stand, ring support
Balance, 0.001-g precision	Triangle, pipe stem
Bunsen burner	Wire gauze
Crucible and cover	

NO

Part 3

Aluminum potassium sulfate, 1 g

Aspirator

Balance, 0.001-g precision

Beaker, 250-mL, 2

Bunsen burner (or hot plate)

~~Filter flask and adapter~~ (or funnel and fine-grained filter paper)~~Gooch crucible with fiber-glass filter pad~~

Graduated cylinder, 50-mL

Ring stand, ring

Rubber policeman

Stirring rod

Wash bottle with distilled water

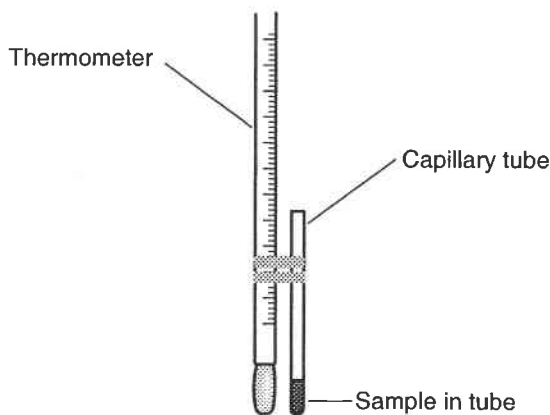
Watch glass

Safety Precautions

Barium nitrate solution is toxic by ingestion. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Thoroughly wash hands with soap and water before leaving the laboratory.

Procedure**Part 1. Melting Point Determination of Alum**

1. Using a mortar and pestle, pulverize a small amount (about 0.5 g) of dry alum.
2. Pack the alum in a capillary tube to a depth of about 0.5 cm. To get the alum into the capillary tube, push the open end of the capillary tube down into a small pile of alum powder.
3. To pack the alum tightly at the closed end of the capillary tube, turn the tube so the open end is up, and bounce the bottom of the tube on the desk top.
4. Fasten the capillary tube to a thermometer with a rubber band. The alum should be level with the thermometer bulb (see Figure 1).



Experimental Setup

Figure 1.

5. Using a universal clamp and cork stopper (or split rubber stopper), fasten the thermometer to a ring stand.

- Immerse the bottom of the capillary and thermometer in a beaker of water (or a Thiele melting point tube filled with water) and heat (Figure 2). If using a beaker, stir the water to maintain an even distribution of temperature. The water bath may be heated rapidly in the beginning, but as the temperature approaches the melting point of alum, the water bath should be heated more slowly in order to get an accurate temperature reading of the melting point.
- Record the temperature range at which the alum melts (the white powder will turn to a colorless liquid) in the Part 1 Data Table.
- Repeat the melting point determination, using a fresh sample of alum and a new capillary tube.

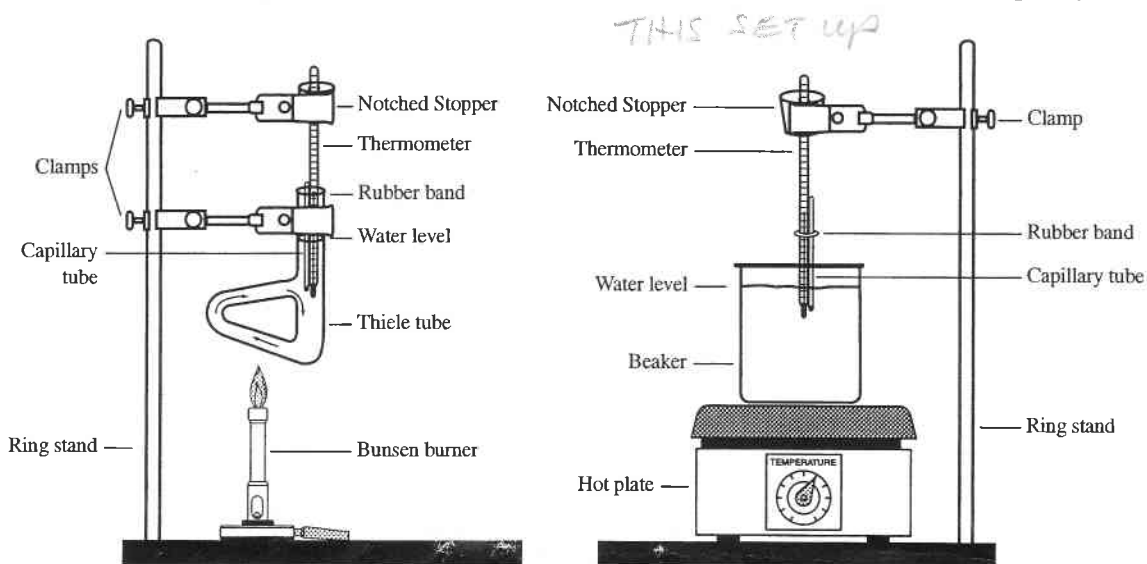


Figure 2.

Part 2. Determination of the Water of Hydration in Alum Crystals

- Set up a Bunsen burner on a ring stand beneath a ring clamp holding a clay pipestem triangle. (See Figure 3.) Do NOT light the Bunsen burner.
- Adjust the height of the ring clamp so that the bottom of a crucible sitting in the clay triangle is about 1 cm above the burner. This will ensure that the crucible will be in the hottest part of the flame when the Bunsen burner is lit.
- Place a crucible with a cover in the clay triangle and heat over a burner flame until the crucible is red hot.
- Turn off the gas source and remove the burner.

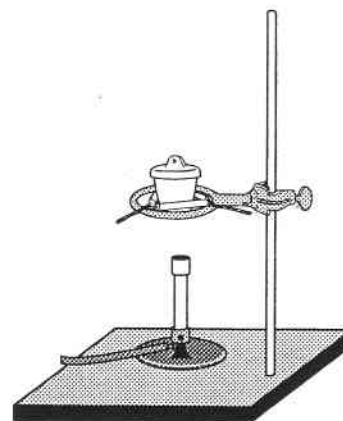


Figure 3.

- Using tongs, remove the crucible cover and place it on a wire gauze on the bench top. With the tongs, remove the crucible from the clay triangle and place it on the wire gauze as well. (See Figure 4.)
- Allow the crucible and its cover to cool completely on the wire gauze for at least 10 minutes.
- Find their mass using an analytical balance. Handle with tongs or forceps to avoid getting fingerprints on the crucible and lid.
- Record their mass in the Part 2 Data Table.
- Now add about 2 g of alum crystals to the crucible. Weigh the crucible, cover, and crystals and record their combined mass in the Part 2 Data Table.

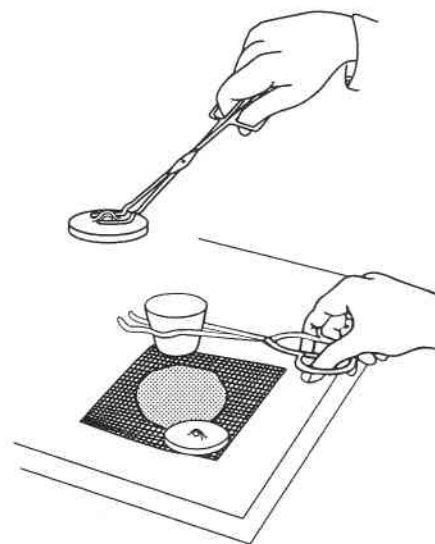


Figure 4.

- Set the crucible at an angle in a triangle held in a ring on a ring stand. Cover the crucible loosely with the crucible cover, and heat very gently. The alum crystals will melt, and the water of hydration will evaporate. It is important that the escaping vapor does not carry any of the anhydrous alum along with it, so be sure that the crystals are heated very gently at first (Figure 5).

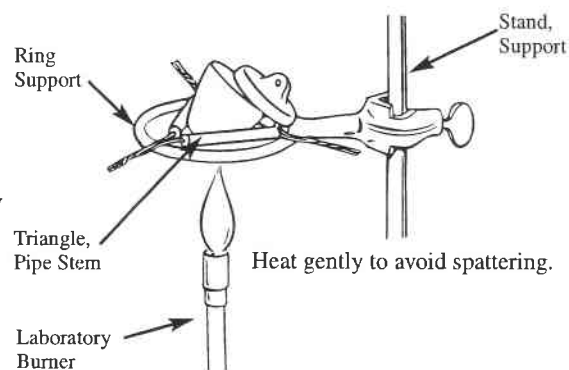


Figure 5.

- After the bubbling has stopped, heat the sample more strongly for an additional five minutes.
- Turn off the gas source and remove the burner.
- Using tongs, remove the crucible cover and place it on a wire gauze on the bench top. With the tongs, remove the crucible from the clay triangle and place it on the wire gauze as well. (See Figure 4.)
- Allow the crucible and its cover to cool completely on the wire gauze for at least 10 minutes.
- Measure and record the mass of the crucible, cover, and anhydrous alum.
- Repeat the drying procedure until constant mass is obtained.
- Record the final mass of the crucible, cover, and anhydrous alum in the Part 2 Data Table.
- Dispose of the anhydrous alum according to your instructor's directions. Carefully clean the crucible and crucible cover.

Sample Data

yes **Part 1. Data Table**

	Trial #1	Trial #2
Measured melting point	°C	°C
Literature melting point	°C	°C

yes **Part 2. Data Table**

Mass of crucible and cover	g
Mass of crucible, cover, and alum crystals	g
Mass of alum crystals	g
Mass of crucible, cover, and alum after heating #1	g
Mass of crucible, cover, and alum after heating #2	g
Mass of water driven off	g
Mass of anhydrous alum, $\text{AlK}(\text{SO}_4)_2$	g
Moles H_2O	
Moles $\text{AlK}(\text{SO}_4)_2$	
Mole ratio; moles H_2O /moles $\text{AlK}(\text{SO}_4)_2$	

NO **Part 3. Data Table**

Mass of alum	g
Mass of dry Gooch crucible <i>Filter paper</i>	g
Mass of Gooch crucible plus barium sulfate <i>FILTER PAPER</i>	g
Mass of barium sulfate	g
Mass of sulfate in precipitate	g
Experimental percent sulfate in alum	%
Theoretical percent sulfate in alum	%

Calculations and Analysis:

Part 1

1. Find the literature value for the melting point of aluminum potassium sulfate and enter this value in the Part 1 Data Table. What was your percent error¹ from the literature value?

Part 2

1. From the mass of anhydrous alum remaining in the crucible after heating and its formula, $\text{AlK}(\text{SO}_4)_2$, calculate the moles of anhydrous alum in the original sample. Enter its value in the Part 2 Data Table.

2. From the mass of water driven off from the sample and the molar mass of water, calculate the *moles* of water in the original sample. Enter this value in the Part 2 Data Table.

3. Calculate the mole ratio of water to anhydrous alum in the sample. Record this value in the Part 2 Data Table.

Post-Lab Questions:

1. Why must objects be cooled before their mass is determined on a sensitive balance?
2. If during the heating process, alum spattered out of the crucible, would the final value for waters of hydration be too high or too low. **Explain** using math or logic.
3. Why did you repeat the drying process several times in the determination of waters of hydration?

$$^1 \text{ Percent Error} = \frac{\text{Experimental} - \text{Actual}}{\text{Actual}} \times 100$$

Name: _____ Period: _____

Rubric for Grading Lab
Analysis of Alum

Points Earned	Points Possible	
	2	Lab is listed with page number in Table of Contents
		The following are copied neatly and completely and with a heading
	2	Title
	3	Experimental Overview
	3	Materials Needed
	2	Heading Procedures: See Flinn Publication number 6354A
	5	Data Table Part 1: Completely filled out
	5	Melting Point of Alum Trial #2 (correct range)
		Melting Point of Alum Trial #2 outside of range (3 points)
	5	Data Table Part 2: Completely filled out
	5	Mole Ratio of water to Alum (correct range)
		Mole Ratio of water to Alum outside of range (3 points)
	2	Heading Calculations and Analysis
		Shows Calculations for:
	3	Part 1 Percent Error for melting point of Alum
	3	Part 2. 1
	3	Part 2. 2
	3	Part 2. 3
	2	Heading Post-Lab Questions
	3	Question 1 addresses hot air currents, etc.
	6	Question 2 addresses whether nH_2O will be to high or low and why
	3	Question 3
	8	Neatness
	68	Total Points

Grader's Name: _____