Alum Lab Part 1: Synthesis of Alum, KAl(SO₄)₂•12H₂O

In this experiment the ionic compound, potassium aluminum sulfate (KAl(SO₄)₂•12H₂O), will be prepared from a water solution that contains K⁺, Al³⁺ and SO₄²⁻ (potassium, aluminum, and sulfate ions, respectively). The aluminum ions will be formed by oxidizing aluminum from aluminum foil. The "double salt" potassium aluminum sulfate dodecahydrate is commonly referred to as alum. Many combinations of mono- and tri-positive cations yield crystals of the same stoichiometry and structure, and alum is a general name for this type of compound. For example, there is chrome alum, KCr(SO₄)₂•12H₂O, which is a deep purple color, as well as alums where either sodium or ammonium ions are present instead of the potassium ion. The crystals are usually in the form of octahedra.

If an aqueous solution which contains [Al(H₂O)₆]³⁺ ions, K⁺ ions and SO₄²⁻ ions is allowed to evaporate, the compound KAl(SO₄)₂•12H₂O will crystallize. Within the alum crystal, six waters of hydration are bonded directly to the aluminum ion to give [Al(H₂O)₆]³⁺ ions, while the other six surround the K⁺ ion.

Alum crystals of great purity are easily prepared. Because of this purity, alum is useful in the dyeing of cloth, where the alum acts as a source of Al³⁺ ions which are not contaminated with Fe³⁺. The Al³⁺ is precipitated on the cloth as aluminum hydroxide which acts as a binding agent for dyes. It is necessary that no Fe³⁺ be present in order to produce clear colors.

Aluminum is considered a reactive metal, but because its surface is usually protected by a thin film of aluminum oxide, it reacts only slowly with acids. It does, however, dissolve quickly in basic solutions. Excess hydroxide ion converts the aluminum to the tetrahydroxoaluminate(III) ion, [Al(OH)₄]⁻. When acid is slowly added to this ion, white, gelatinous aluminum hydroxide (Al(OH)₃ precipitates. Continued addition in solution as the of acid causes the hydroxide ions to be completely neutralized, and the aluminum exists hydrated ion [Al(H₂O)₆]³⁺. Aluminum hydroxide is considered to be an "amphoteric" hydroxide because it dissolves in both acids and bases.

Chemicals

- Aluminum foil
- Sulfuric acid, H₂SO₄
- 3M Baking soda, NaHCO₃ (s)
- Potassium hydroxide, KOH, 3 M
- Water-ethanol solution 50% by volume
- Vinegar, dilute HC₂H₃O₂

Equipment

- Beaker, 250 mL Graduated cylinder
- Buchner funnel and filter flask
- Ice bath
- Balance
- Watch glass
- Stirring rod
- Burner, ring stand, ring, wire gauze
- Plastic wrap or Parafilm®
Procedure

Safety Alert

You will be using solutions with high concentrations of sulfuric acid and potassium hydroxide, both of which are highly damaging to skin and eyes. Be careful when handling them. If you spill any on yourself, wash off with lots of water. Neutralize sulfuric acid spills on the counter with baking soda, and neutralize potassium hydroxide spills with vinegar (dilute acetic acid).

When aluminum dissolves in potassium hydroxide solution, hydrogen gas is produced. Make sure that no flames are present. This should be done in a well-ventilated room.

Wear Chemical Splash Goggles and a Chemical-Resistant Apron.

A) Weigh Out and Dissolve the Aluminum.

1. Weigh out about 1 gram of aluminum foil to the nearest centigram. Tear the foil into small pieces and place in a 250 mL beaker.

2. Slowly add 25 mL of 3 M KOH solution. Allow the reaction to proceed until all of the foil is dissolved.

3. Remove any undissolved solids such as carbon particles by filtering the solution through a Buchner funnel while the solution is hot. Rinse the filter paper with a small amount of distilled water.

B) Acidify with Sulfuric Acid

4. At this point the solution contains [Al(OH)₄]⁻ and K⁺ ions, along with excess OH⁻ ions. Cool the solution and then acidify it SLOWLY, with constant stirring, using 35 mL of 3M H₂SO₄. The solution will get very hot because you are adding strong acid to the strongly basic solution. Solid Al(OH)₃ will first precipitate and then dissolve as more H₂SO₄ is added.

5. If a precipitate still remains, filter the solution and discard the solids. You can use vacuum filtration with a Buchner funnel and filter flask to speed up this process.

6. Then boil the solution until water has evaporated to give a volume of about 50 mL of solution. This is a good place to stop if the end of the lab period is near. Cool the solution and cover the beaker with Parafilm® or plastic wrap. Allow it to rest undisturbed until the next period.
C) Crystallize Alum

7. If time permits, cool the solution in an ice bath for 15 minutes, keeping it as motionless as possible. Crystals of alum should grow in the beaker. If no crystals form, scrape the bottom of the beaker with a stirring rod to create a rough place where crystals may begin to grow, or add a seed crystal. If there are still no crystals, reheat the solution until more water has evaporated and then cool again. Rapid cooling in an ice bath causes very small crystals to grow; slow overnight cooling allows the formation of larger crystals. Collect the alum crystals by vacuum filtration.

8. Wash the crystals with 50 mL of a 50% by volume water and ethanol mixture, in which alum crystals are not very soluble.

9. Allow the crystals to dry at room temperature.

10. Determine the mass of the alum. Calculate the theoretical yield of alum assuming that aluminum was the limiting reactant and that the foil was 100% aluminum, and calculate your percent yield.

11. Verify that your crystals are alum by performing Alum Lab Part 2, "Analysis of Alum."

Disposal

Pour the filtrate into a beaker which your instructor will provide for disposal. See the Flinn Chemical Catalog/Reference Manual, suggested disposal method #24b. See the appendix.

Pre-Lab Questions

1. What is alum?

2. What is a hydrated crystal?

3. What is meant by the term amphoteric?

4. If one carried out a reaction to synthesize KAl(SO₄)₂•12H₂O using 1.0 g of potassium metal as the limiting reagent, what would be the maximum mass of alum that could form? Show your calculations.

5. If one carried out the procedure in question 6 and actually obtained 4.5 g of alum, what would be the percent yield? Show your calculations.

6. How do you clean acid and base spills?
Alum Lab Part 2: Synthesis of Alum, KAl(SO₄)₂•12H₂O

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 various tests that can be performed to verify that the compound is the one desired. In the previous experiment, alum crystals, KAl(SO₄)₂•12H₂O, were prepared. In this experiment we will do several tests to determine if the crystals are really alum. Alternatively, you may be given an unknown compound to analyze.

The first and simplest test 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 second test that we can do is to determine the amount of water of hydration present in the alum crystals. Some of the alum is 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 moles water to moles KAl(SO₄)₂ can be calculated and then compared to the correct formula values.

Chemicals

“Alum crystals- KAl(SO₄)₂•12H₂O” – made during Alum Lab Part 1

Equipment Part 1:

- Capillary tube
- Beaker (or Thiele melting point tube)
- Ring stand, ring, wire gauze
- Thermometer
- Rubber band
- Universal clamp

Equipment Part 2:

- Crucible and cover
- Clay triangle
- Crucible tongs

- Cork (or split stopper) to hold thermometer
- Mortar and pestle (or test tube and watch glass)
- Stirring rod
- Bunsen burner
- Ring stand, ring, Bunsen burner
- Triangle support
**Procedure**

*Safety Alert*- Be cautious using flames.

A. Find the Melting Point of Alum

1. Pulverize a small amount (about 0.5 g) of dry alum. Use a mortar and pestle.

2. Pack the alum in a capillary tube to a depth of about 1 cm. To get the alum into the capillary tube, push the open end of the capillary down into a small pile of alum powder. Then turn the tube so the open end is up, and bounce the bottom of the tube on the desk top.

3. Use a rubber band, and with it fasten the capillary tube to a thermometer. The alum should be level with the bulb of the thermometer.

4. Use a thermometer clamp or universal clamp and cork stopper (or split rubber stopper) to fasten the thermometer to a ring stand.

5. Immerse the bottom of the capillary and thermometer in a beaker of water (or a Thiele melting point tube filled with water) and heat. If using a beaker, you must stir the water to maintain an even distribution of temperature. You may heat rapidly in the beginning but as you get close to the melting point, heat very slowly in order to get an accurate value.

6. Record the temperature at which the alum melts (the white powder will become clear). If you wish to repeat the melting procedure, use both a new sample and a new capillary tube.

7. Find the published value for the melting point of alum, and compare the experimental and published values.
B) Determine the amount of Water of Hydration in Alum Crystals

1. Set up a Bunsen burner on a ring stand beneath a ring clamp holding a clay pipe stem triangle. Do NOT light the Bunsen burner.

2. 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.

3. Place a crucible with a cover in the clay triangle and heat over a burner flame until the crucible is red hot.

4. Turn off the gas source and remove the burner.

5. When cooled determine the mass of the crucible and cover. Handle with tongs or forceps to avoid getting fingerprints on them.

6. Now add about 2 g of alum crystals to the crucible. Accurately determine the mass of the crucible, cover, and crystals.

7. Set the crucible at an angle in a triangle held in a ring on a ring stand, cover loosely with the crucible cover, and heat very gently. The alum crystals will melt, and the water of hydration will vaporize. 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. After the vapor is apparently driven off, heat more strongly for five minutes.

8. Cool, and find the mass of the crucible, cover, and anhydrous alum.
9. Calculate the mass of the anhydrous alum and the mass of the water that was driven off.

10. Determine the number of moles of the anhydrous alum and the number of moles of water that was driven off.

11. Determine the mole ratio of anhydrous alum to the water that was driven off. Compare to Alum.

Pre-Lab Questions

1. When finding 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 $\text{Na}_2\text{CO}_3\cdot12\text{H}_2\text{O}$, where $X$ is the number of moles of $\text{H}_2\text{O}$ per mole of $\text{Na}_2\text{CO}_3$. When a 2.123 g sample of washing soda is heated at 130°C, all of the water of hydration is lost, leaving 0.789g of anhydrous sodium carbonate. Calculate the value of $X$.

3. During the heating of this experiment should the crucible be closed or open? Should this be the same during cooling? Explain.
Post Lab Questions

1. What is a "synthesis" reaction?
2. Why should you NOT expect a 100% yield of crystals?
3. How does the solubility of alum in water change with temperature?
4. Why should you NOT wash the crystals with pure water?
5. What do your crystals look like?
6. What is the shape of an octahedron?
7. Show how you obtained your theoretical and percent yields.
8. Why must objects be cooled before their mass is found on a sensitive balance?
9. Comment on the results of the different tests you used to verify that the substance tested was alum.
10. What other tests could be made to verify the composition of alum?