Preparation and Analysis of Potassium Alum

This experiment describes the preparation and analysis of potassium alum, commonly known as simply alum. Starting from very familiar materials – aluminum foil, potassium hydroxide, and sulfuric acid—you will produce beautiful crystals which have a number of practical uses. Alum was first used as a mordant (fixing agent) for dyed textiles around 1000 BCE. Today, its main use is as a coagulant in water purification. Additional applications include paper sizing (changing surface properties to help ink adhere), leather tanning, pickle making (available on the spice shelf; makes crisper pickles) and medicinal use as an astringent (styptic pencil to stop bleeding).

Every compound has a unique set of chemical and physical properties. In this experiment, you will determine the melting point and the number of moles of water of hydration in potassium alum.

In the first step of the synthesis, aluminum metal rapidly reacts exothermically with KOH solution in a redox reaction:

\[
2 \text{Al}(s) + 2 \text{KOH}(aq) + 6 \text{H}_2\text{O}(l) \rightarrow 2\text{KAl(OH)}_4(aq) + 3\text{H}_2(g)
\]

or the net ionic equation

\[
2 \text{Al}(s) + 2 \text{OH}^-(aq) + 6 \text{H}_2\text{O}(l) \rightarrow 2 \text{Al(OH)}_4^-(aq) + 3 \text{H}_2(g)
\]

Sulfuric acid is now added and two sequential reactions occur. Initially, before addition of all the acid, the reaction is

\[
2 \text{KAl(OH)}_4(aq) + \text{H}_2\text{SO}_4(aq) \rightarrow 2 \text{Al(OH)}_3(s) + \text{K}_2\text{SO}_4(aq) + 2 \text{H}_2\text{O}(l)
\]

or the net ionic equation

\[
\text{Al(OH)}_4^-(aq) + \text{H}^+(aq) \rightarrow \text{Al(OH)}_3(s) + \text{H}_2\text{O}(l)
\]

The reaction above is an acid-base reaction in which the H\(^+\) ions from sulfuric acid neutralize the base \(\text{Al(OH)}_4^-\) to give a thick, white, gelatinous precipitate of aluminum hydroxide, \(\text{Al(OH)}_3\). As more sulfuric acid is added, the precipitate of \(\text{Al(OH)}_3\) dissolves and reacts further

\[
2 \text{Al(OH)}_3(s) + 3\text{H}_2\text{SO}_4(aq) \rightarrow \text{Al}_2(\text{SO}_4)_{3}(aq) + 6 \text{H}_2\text{O}(l)
\]

or the net ionic equation

\[
\text{Al(OH)}_3(s) + \text{H}^+(aq) \rightarrow \text{Al}^{3+}(aq) + 3 \text{H}_2\text{O}(l)
\]

to give aluminum ions, \(\text{Al}^{3+}\), in solution. The solution at this point contains \(\text{Al}^{3+}\) ions, \(\text{K}^+\) ions, and \(\text{SO}_4^{2-}\) ions. On cooling, crystals of hydrated potassium aluminum sulfate, \(\text{KAl(SO}_4)_2 \cdot n\text{H}_2\text{O}(s)\), or alum, are slowly deposited.

Finally, the crystals of alum are removed from the solution by vacuum filtration and washed with an alcohol/water mixture. This wash liquid removes any contamination from the crystals but does not dissolve them. It also helps to dry the crystals quickly, because alcohol is more volatile than water.

PROCEDURE

Part 1. Synthesis of Alum

1. **Prepare the Aluminum Sample**. Cut approximately 0.5 g aluminum foil into small pieces. Tare a 100-mL beaker and mass the aluminum pieces, record.

2. **Dissolve the Aluminum Pieces**. This step must be done in a fume hood; if a fume hood is not available, connect a large plastic funnel to a well-functioning aspirator and place the funnel directly over the reaction flask. Add 10-12 mL of 4 \(M\) KOH to the aluminum pieces (Caution: Wear safety glasses; do not splatter the solution, KOH is caustic) and swirl the reaction mixture. Some brands of aluminum foil will react immediately; for others you may need to warm the beaker gently with a hot plate to initiate the reaction. As the reaction proceeds hydrogen gas is being evolved as is evidenced by the "fizzing" at the edges of the aluminum pieces.

The dissolution of the aluminum pieces may take up to 20 minutes—it is important to maintain the solution at a level that is one-half to three-fourths of its original volume by adding small portions of deionized water during the dissolution process.
3. **Filter the Reaction Mixture.** When no further reaction is evident, return the reaction mixture to the laboratory desk. Filter the warm reaction mixture to remove the insoluble impurities. If solid particles appear in the filtrate, repeat the filtration. Rinse the filter with 2-3 mL of deionized water.

4. **Allow the Formation of Aluminum Hydroxide.** Allow the clear solution (the filtrate) to cool in the 100-mL beaker. While stirring, slowly add, in 5-mL increments (because the reaction is exothermic!), approximately 15 mL of 6 M H₂SO₄. (Caution: Avoid skin contact!).

5. **Dissolve the Aluminum Hydroxide.** If the solution shows evidence of the white, gelatinous Al(OH)₃ precipitate in the acidified filtrate, gently heat the mixture until the Al(OH)₃ dissolves and you have a clear solution.

6. **Crystallize the Alum.** Remove the solution from the heat. Cool the solution in an ice bath. Alum crystals should form within 20 minutes. If crystals do not form, use a hot plate to gently reduce the volume by one-half (do not boil!) and return to the ice bath. For larger crystals and a higher yield, allow the crystallization process to continue until the next laboratory period. Alternatively, simply set aside overnight.

7. **Isolate and Wash the Alum Crystals.** Vacuum filter the alum crystals from the solution. Wash the crystals on the filter paper with two (cooled-to-ice temperature) 5-mL portions of a 50% (by volume) ethanol-water solution. (The alum crystals are marginally soluble in 50% by volume ethanol-water solution). Maintain the suction until the crystals appear dry. Determine the mass of the crystals. Calculate the percent yield.

8. **CLEANUP** with soap and water.

**Part 2. Determine the Melting Point of Alum**

1. Look up the actual value for the melting point of potassium alum in the CRC Handbook.
2. Using a mortar and pestle, pulverize a small amount (about 0.1 g) of dry alum.
3. 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. Pack by tamping.
4. Heat the melting point apparatus at the appropriate setting for the melting point of alum. Observing through the sight glass, record the melting point range of your sample: where the first droplets of liquid appear until the entire sample is melted.
5. Cool the heating block with a cold metal slug for the next group of students.

**Part 3. Determination of the Water of Hydration in Alum Crystals**

1. Clean your crucible and cover by placing a few drops of 1 M NH₃ solution in the empty crucible and scrubbing with a paper towel. Rinse the crucible with distilled water and place the empty crucible on the porcelain triangle supported by a ring and ring stand.
2. Heat crucible and a cover until red hot. Allow to cool before using.
3. Determine the waters of hydration of potassium alum. Be careful to avoid spattering when you start heating.
4. Dispose of the anhydrous alum in the trash. Return the clean crucible and cover to the teacher.

**Calculations and Analysis**

**Part 1.** Calculate the percent yield of potassium alum you obtained, based on the amount of aluminum used.

**Part 2.** Compare the literature value for the melting point of potassium alum with your experimental value. If your melting point is off by more than 5°C, suggest an explanation.

**Part 3.** Calculate the mole ratio of water to anhydrous alum in the sample, and thus the formula for potassium alum. If your waters of hydration are off by more than 10%, suggest an explanation.