# **Chemical Synthesis of Alum**

# Introduction

Alum is an example of a hydrated ionic compound. The two cations are the potassium and aluminium ions, K + and Al 3+; the anion is the sulfate ion, SO4 2-. A hydrate is a crystal that includes water molecules in its structure. In the case of alum, it is a dodecahydrate, with 12 water molecules for each formula unit. Therefore, the empirical formula of alum is K Al (SO4)2 · 12H2O.

Alum is prepared in this experiment through a series of reactions. The first step is to dissolve aluminum in a concentrated potassium hydroxide solution, where the aluminum atom forms a complex ion with hydroxide.

2 Al(s) + 2 OH−(aq) + 6 H2O(l) → 2 Al(OH)4−(aq) + 3 H2(g)

When sulfuric acid is added to the solution, the acid neutralizes the hydroxide ions, freeing up the aluminum ion. When precipitated, the product is alum.

K+(aq) + Al(OH)4−(aq) + 2 H2SO4(aq) + 8 H2O(l) → K Al (SO4)2 · 12H2O(s)

# Materials and Hazards

* Aluminum foil
* 1.4 M Potassium Hydroxide, KOH Corrosive, Mutagenic, Toxic
* 9 M Sulfuric Acid, H2SO4 Corrosive, Carcinogenic, Poisonous
* Ethanol Flammable
* Scissors
* 150 mL, 250 mL and 600 mL beakers
* 10 mL and 50 mL graduated cylinders
* Glass funnel
* Glass stirring rod
* Ice
* Thermometer
* Plastic pipet
* Filter paper for gravity filtration
* Filter paper for vacuum filtration
* Filter flask
* Buchner funnel, hose, and rubber gasket

# Procedure

Step 1

1. Cut a piece of aluminum foil into smaller pieces.
2. Obtain a mass of aluminum between 0.1 and 0.2 g, to the closest 0.001 g.
3. Record the weight of the metal on the Report Sheet and place the aluminum in a 150 mL beaker.

Step 2

1. **In a fume hood**, *slowly* add 25 mL of 1.4 M potassium hydroxide, KOH, to the aluminum sample.

2. Cover the beaker with a watch glass and warm the solution on a hot plate set to about ¼ power. Heat the solution without boiling and swirl or stir it occasionally.

The aluminum should dissolve in about 20 minutes. Ignore any black solid that may be suspended in the liquid. The reaction is complete when there is no more bubbling and no visible pieces of aluminum.

Step 3

While the aluminum is dissolving, set up a funnel supported by a ring. Obtain a piece of filter paper (large size) and fold it as shown by the instructor. Place the filter paper in the funnel and wet it with water from a wash bottle, discarding the water.

Step 4

1. In the hood, add 10 mL of 9 M sulfuric acid to the solution *quickly but carefully* with stirring. The reaction is exothermic so the solution will get hotter as the acid is added.
2. If there are white crystals in the liquid, heat the mixture gently on a hot plate to dissolve them.
3. Take the beaker to the workstation using the method demonstrated by your instructor. Filter the solution using the apparatus in Step 3 into a 250 mL beaker.

Step 5

1. Fill a 600-mL beaker halfway with ice, adding just enough tap water to barely cover it. Place a thermometer in the beaker but do not use the thermometer for stirring. Cool the beaker in the ice bath with regular stirring (with the stirring rod) for 20 minutes or until the temperature reaches 6°C.
2. If no crystals form, induce crystallization by scratching the bottom of the beaker with a glass stirring rod *or* create a seed crystal by wetting the stirring rod with the alum solution, taking it out and blowing on the tip to dry it until crystals form, and then dipping the rod back into the solution.

Step 6

1. Weigh a piece of filter paper (small size) suitable for vacuum filtration.
2. Prepare a Buchner funnel for vacuum filtration and lay the filter paper in its base.
3. Filter the mixture using vacuum filtration. Use the ethanol bottle to rinse any remaining crystals out of the beaker and into the funnel. Wash the crystals with additional ethanol to remove excess water from the crystals. Allow the aspirator to pull air through the crystals for about 5 minutes.
4. Turn off filtration apparatus and place the filter paper and product on a watch glass. Place it in a drying oven set to 700 C for 5 minutes.
5. Cool the filter paper and product and weigh it on a balance. Record the mass of the paper and product on the Report Sheet to the closest 0.001 g.

# Data and Results

Determine to theoretical yield of alum from the initial mass of aluminum, the balance equation, and the molar mass of alum.

Determine the percent yield from the theoretical yield and the actual yield of alum.

Clean Up

Pour the liquid filtrate from the vacuum flask into the designated waste container and put the alum in its designated waste container. Wash all glassware thoroughly before putting it away. Leave vacuum filtration set up but empty the liquid from the flask into waste but everything else away.