EXPERIMENT 3
SYNTHESIS OF AN ALUM
$KAl(SO_4)_2 \cdot 12H_2O$

INTRODUCTION

Potassium aluminum sulfate dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$, belongs to a class of inorganic compounds called alums (which is accented on the first syllable). The composition of alum is represented by the general formula $M^+M^{3+}(SO_4)_2 \cdot 12H_2O$. In this formula, M^+ is a monoatomic ion such as Na^+, K^+, Tl^+, or Ag^+; or is NH_4^+. Although the name *alum* sounds as if the compound must contain aluminum, the M^{3+} might be from any of several main-group metals (p block elements) and transition metals (d block elements) that form triply charged ions: Al, Ti, V, Cr, Mn, Fe, Co, Ga, In, and Ir. Some examples are $NaAl(SO_4)_2 \cdot 12H_2O$, which is a component of some baking powders; $NH_4Al(SO_4)_2 \cdot 12H_2O$, which is used in dyeing textiles; and $KCr(SO_4)_2 \cdot 12H_2O$, which is used in tanning leather.

The term *double salt* also describes $KAl(SO_4)_2 \cdot 12H_2O$. A double salt is an ionic compound that contains two different cations or two different anions. In aqueous solution, double salts give positive tests for all three of the ions they contain. $KAl(SO_4)_2 \cdot 12H_2O$ would give positive tests for K^+, Al^{3+}, and SO_4^{2-}.

Hydrate is another term that describes $KAl(SO_4)_2 \cdot 12H_2O$. A hydrate is a substance that contains a specific number of water molecules per formula unit in its solid form. Hydration is common with ionic compounds, for example $Na_2SO_4 \cdot 10H_2O$ and $CoCl_2 \cdot 6H_2O$. **The water of hydration is included when calculating the compound's formula mass**. The water molecules are part of the crystal of a compound along with cations and anions, but they can break out of the crystal when it is heated. This dehydration might occur at a specific temperature characteristic of the particular compound. If the compound is heated in a relatively closed container, the escaping water might dissolve the remaining salt forming a liquid solution. Consequently, this combination of dehydration and dissolving looks like melting; the temperature range at which it occurs (the solid-to-liquid transition) can be measured in the same way that a melting point is measured.

Reactions Involved in the Synthesis

Metals dissolve in water or aqueous solutions only by reacting with water or some other component of the solution. The reaction converts the metal to a positive ion. Aluminum dissolves in acids by reacting with H_3O^+ and dissolves in bases by reacting with OH^-. The reaction you will use to dissolve aluminum involves OH^- from KOH:

$$2\ Al(s) + 2\ OH^-(aq) + 6\ H_2O \rightarrow 2\ Al(OH)_4^-(aq) + 3\ H_2(g) \qquad (3\text{-}1)$$

Next, you will add sulfuric acid to the solution containing $Al(OH)_4^-$. As H_3O^+ reacts with $Al(OH)_4^-$, insoluble $Al(OH)_3$ forms and precipitates. As more acid is added, the precipitate $Al(OH)_3$ dissolves:

$$Al(OH)_4^-(aq) + H_3O^+(aq) \rightarrow Al(OH)_3(s) + 2\ H_2O(l) \qquad (3\text{-}2)$$

$$Al(OH)_3(s) + 3\ H_3O^+(aq) \rightarrow Al^{3+}(aq) + 6\ H_2O(l) \qquad (3\text{-}3)$$

The product forms and precipitates when the solution containing Al^{3+}, K^+ (from KOH), and SO_4^{2-} (from H_2SO_4) is cooled:

$$K^+(aq) + Al^{3+}(aq) + 2\ SO_4^{2-}(aq) + 12\ H_2O\ (l) \rightarrow KAl(SO_4)_2{\cdot}12\ H_2O(s) \qquad (3\text{-}4)$$

EQUIPMENT NEEDED

balance
beakers
Büchner funnel
hot plate
clamp
conical funnel
Erlenmeyer flasks
filter flask
filter paper—12.5cm and 7.0cm
graduated cylinder
melting point capillary tubes
ring stand
rubber O-ring
slit stopper
temperature probe
spatula
watch glass
glass stirring rod

CHEMICALS NEEDED

isopropyl alcohol
Al foil
ice
6 M H_2SO_4; sulfuric acid
3M KOH; potassium hydroxide
thymol blue solution

PROCEDURE

Dissolving Al by Reaction with KOH

1. Weigh a piece of aluminum foil on the laboratory balance. Remove some Al from it or add more Al to it until you have ~0.5–0.6 grams; record the mass. Tear the Al into pieces about the size of large postage stamps and put these pieces into an Erlenmeyer flask.
2. Take a graduated cylinder to the hood and measure out ~20mL of 3M KOH solution ***(Caution—caustic material!)***. Take the graduated cylinder back to your bench area and pour the 3M KOH solution into the flask containing the aluminum foil. Within a few minutes the Al and KOH will begin to react producing hydrogen; this reaction will become progressively more vigorous. **Be careful when handling the flask; the reaction is so exothermic that the entire flask, including the neck, will become extremely hot.** After the obvious evolution of hydrogen ceases, let the flask stand for a few minutes more to ensure complete reaction of the Al.
3. If the solution in the flask is *absolutely clear*, proceed to the next step in the synthesis. If the solution is not absolutely clear, filter it by gravity filtration. Collect the filtrate in another Erlenmeyer flask. Discard the filter paper in the regular trash container.

Converting $Al(OH)_4^-$ To Al^{3+} by Reaction with H_2SO_4

4. Take a graduated cylinder to the hood and measure out ~16.0mL of 6M H_2SO_4 solution (***Caution—caustic material!)***. Take the graduated cylinder back to your bench area. To the clear filtered reaction solution, add the 6M H_2SO_4 solution a few milliliters at a time. After each addition, swirl the flask to mix the contents. Initially, $Al(OH)_3$ will precipitate; then some of it will dissolve as more H_2SO_4 is added. If solid sticks to the inside wall of the flask, wash it down with a few drops of H_2SO_4. After you have added all the H_2SO_4, gently heat the flask for several minutes; have the liquid *barely bubbling*. If the amount of H_2SO_4 added to the reaction mixture was sufficient for the amounts of Al and KOH you used, all of the solid in the flask should dissolve.
5. While the reaction mixture is heating, fill an 800 mL beaker with ice and add just enough tap water to cover the ice. Wash out the unused Erlenmeyer flask, fill it with distilled water, and place it in the ice water bath. This will be your wash water.

Precipitating $KAl(SO_4)_2{\cdot}12H_2O$ by Cooling

6. Remove the flask of distilled water from the ice water bath and replace it with the flask containing the reaction mixture. Occasionally stir the solution with a glass rod. After crystals of the product begin to form, cool the mixture for at least another ten minutes; continue to stir it occasionally. While you wait for the product to precipitate, set up the equipment for filtration.

Collecting, Washing, and Drying $KAl(SO_4)_2{\cdot}12H_2O$.

7. Set up a Büchner funnel apparatus. Clamp the filter flask to a ring stand and connect the sidearm of the flask to the vacuum line with a long piece of pressure tubing. Place the funnel on top of the flask, and insert a piece of filter paper. Open the vacuum line so that it is pulling air through the funnel. Wet the filter paper in the funnel with some water from a beaker until it forms a seal with the base of the funnel.
8. Remove the flask containing the product from the ice water bath and replace it with the flask containing the wash water. Swirl the flask with your product so that the solid is dispersed in the solution, then pour it into the funnel. Wash any remaining solid left in the flask into the funnel with a small amount of *ice-cold* water. The water must be as cold as possible; since the product is slightly soluble in water, using cold water ensures that the amount of product that is dissolved away is minimized. When no more drops of water are seen coming out of the tip of the funnel, turn off the vacuum.
9. The *washing* of the precipitate is done to purify the product by removing the excess ions present in the starting materials from the product. This step takes advantage of the differing solubility properties of the impurities and the product; the ionic impurities are all readily soluble in water, while the alum is only slightly soluble in water. One of the ionic impurities is hydronium ion; we can monitor the progress of the washing by using an indicator to detect the presence of hydronium ion. Put 2 or 3 drops of distilled water into a small test tube, add one drop of thymol blue solution, then remove the funnel and tubing from the filter flask and transfer a few drops of the filtrate (the liquid in the filter flask) into the thymol blue solution with a disposable pipet. The first filtrate that you collect should give a pink color with the indicator showing that an excess of acid was used in the reaction. After testing this first filtrate, keep the test tube of indicator and filtrate to use to make color comparisons with subsequent washings.