

CHEM 231

Experiment 5

Synthesis of Alum

Alum is a solid ionic compound with many uses. It is used as astringent to prevent bleeding from small cuts, as an ingredient in deodorants, as an ingredient in baking powders, and as a preservative used in pickling.

The formula of alum is $KAl(SO_4)_2 \cdot 12H_2O$ and the standard name is potassium aluminum sulfate. It is an unusual ionic compound in that it includes two positive ions, K^+ and Al^{3+} . It also includes 12 water molecules in each formula unit which remain in the solid when it is precipitated from a water solution. Such water molecules are referred to as "waters of hydration" and ionic compounds which incorporate them into their solid structures are called "hydrates." Many solid ionic compounds exist as hydrates. It is important to include these water molecules in the formula weight of the compound when doing stoichiometric calculations.

The synthesis of alum in this experiment is carried out beginning with aluminum from aluminum cans. This is not how alum is produced commercially. Aluminum does not occur in nature as the native metal and it is very costly to produce. Thus, the commercial production of alum begins with minerals containing aluminum in its oxidized state rather than elemental aluminum.

The purpose of the experiment is to introduce some common laboratory techniques used in chemical synthesis, to demonstrate a synthesis of a useful substance, and to apply the concepts of limiting reactant and reaction yield.

Preliminaries

What are the ions occurring in alum? Confirm that the ion charges in the empirical formula add up to zero.

Find the formula weight of alum. Be certain to include the waters of hydration.

Procedure

Step 1: Oxidation of aluminum

Cut a piece of aluminum about 5 cm square from an aluminum can. This aluminum has a label on the outside as well as an invisible plastic coating on the inside. Clean these off using a piece of sandpaper. Weigh the aluminum piece and trim it with a scissors until its mass is approximately 0.5 g. Obtain a precise mass of this aluminum piece on an analytical balance.

This mass is very important because aluminum is the limiting reactant and determines the maximum possible yield of the alum product.

Cut the aluminum into small pieces with a scissors and place in a 150 mL beaker. Obtain 25 mL of 1.4 M KOH solution. Working in the hood, add this solution to the beaker containing the aluminum. Place the beaker on a hot plate set at low heat. Warm the solution, but do not allow it to boil. With occasional stirring, all the aluminum should react in about 20 min.

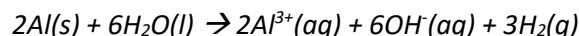
While the aluminum is dissolving, set up long-stem glass funnel using a funnel support on a ring stand. Fold a piece of filter paper into quarters, and place it in the funnel as demonstrated by your instructor. Wet the filter paper with a small amount of distilled water. The stem of the funnel should empty into a 150-mL beaker.

The reaction of the aluminum is complete when no aluminum remains in the beaker and bubbling has ceased. The solution may have a dirty "cloudiness" due to undissolved solids. This is due to impurities which are to be removed by filtration.

When the reaction is complete, pour the reaction mixture through the funnel. Rinse the reaction beaker twice with 2 mL distilled water adding each rinse to the filter paper. Collect and save this liquid filtrate. Rinse the residue on the filter paper with 2-3 mL of distilled water, joining this rinse to the filtrate. Save the filtrate for the next step. The filtrate should be a clear solution which contains the desired product of the reaction.

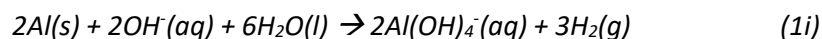
Background information

The primary chemical change happening in the first step is the oxidation of aluminum to aluminum ion by water. Aluminum, like the alkali metals and alkaline earth metals, is a very active metal which can be oxidized by water. For aluminum, a balanced reaction is:



The situation is actually a bit more complicated than this. First, although aluminum is a very active metal, it does not readily react with water directly. If it did, we could not use aluminum cans the way we do. The reason this does not ordinarily happen is that aluminum metal becomes coated by an oxide layer when it comes in contact with air. This layer is impervious and protects the metal from further oxidation. A strong base like potassium hydroxide will dissolve the oxide layer, exposing the metal and allowing the above reaction to take place.

*Second, in the presence of strong base, the aluminum ion does not exist in isolation but rather as a so-called complex, a central ion surrounded by several other molecules, called **ligands**. The complex formed in this case consists of a central Al^{3+} ion surrounded by 4 hydroxide (OH^{-}) ions. This complex has a net charge of -1. We can rewrite the chemical equation above reflecting the formation of the complex. In doing so, we can use hydroxide ions supplied by the potassium hydroxide solution. The result is the net ionic equation for the first step:*



Finally, if we wish to write a full molecular equation for this step, we include a potassium ion for each negative charge appearing on the each side of the equation. The result is:



Although K^+ is only a spectator ion in this step, in the overall synthesis, it is more than just a spectator. The final solid product incorporates potassium ions and the source of this potassium is the potassium hydroxide used in this step.

Questions

Based on the background information and your observations, answer the following questions before continuing:

Aluminum is oxidized in this step. Which element is reduced?

The balanced reaction for this step shows 8 hydroxide ions as part of two $\text{Al}(\text{OH})_4^-$ complexes. Two of these come from KOH. Where do the other 6 come from?

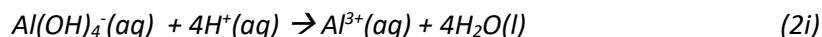
Step 2: Completing the formation of potassium aluminum sulfate

Slowly and carefully add 10 mL of 9M H_2SO_4 to the reaction mixture while stirring. Record all observations, including the formation of a precipitate if any, and any changes in the temperature of the solution.

If there is a precipitate remaining at this point, heat the solution until it dissolves. This is very important. This precipitate is probably **not** your desired product.

Background information

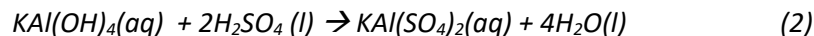
The primary chemical change this step is the neutralization of the hydroxide ions within the complex ions produced in the last step. Each $\text{Al}(\text{OH})_4^-$ ion requires 4 hydrogen ions, leading to the net ionic equation:



If this neutralization occurs in steps, then this reaction produces, in succession, $\text{Al}(\text{OH})_3$, $\text{Al}(\text{OH})_2^+$, $\text{Al}(\text{OH})^{2+}$, and Al^{3+} . The ions are soluble in hot water, but the neutral aluminum hydroxide is not. This is why you may see the appearance of a precipitate which redissolves.

We can turn the net ionic equation for this step into a full molecular equation by rewriting the ion complex ion as the $\text{KAl}(\text{OH})_4$ from the last step, and the hydrogen ions as sulfuric acid, H_2SO_4 . Then the products would include all of the ions present in the final product in the correct proportions: K^+ , Al^{3+} , and SO_4^{2-} , and can be written as $\text{KAl}(\text{SO}_4)_2$. Thus, this step can be regarded

as the completion of the synthesis of potassium aluminum sulfate, although it still exists as separate ions in solution. The full equation is:



Notice that the sulfuric acid not only reacts with the hydroxide ligands but also provides the sulfate which is incorporated into the final product.

Step 3: Precipitation and separation of alum

The desired product is now in solution in the beaker. Although it is soluble in hot water, its solubility decreases with if the solution is cooled.

Allow the 150-mL beaker containing the filtrate to cool. Fill a 600-mL beaker about half full of crushed ice. Place the 150-mL beaker in the ice bath for about 20 min, stirring frequently. You should see crystals of alum form. As the solution is cooling, set up a vacuum filtration apparatus with a Buchner funnel as demonstrated by your instructor.

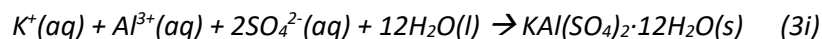
Also while you are waiting for the solution to cool, weigh a clean, dry 150-mL beaker for the purpose of collecting and weighing the final solid product.

Turn on the aspirator or house vacuum. Remove the beaker containing the alum from the ice bath, and stir the contents of the beaker with a stirring rod to loosen all of the crystals. Transfer all of the crystals to the Büchner funnel. A rubber policeman may be useful in this process. Add two 10-mL portions of methanol in succession to wash the excess sulfuric acid out of the crystals. Continue to draw air through the alum for another 10 to 15 min to dry the crystals.

Transfer the dried alum crystals to the weighed beaker. Use a spatula to scrape off any crystals adhering to the filter paper. Reweigh the beaker containing the alum crystals, and record this mass.

Background information

It is very common for an ionic compound to be soluble in hot water but insoluble in cold water. Cooling a solution is a common way to remove such an ionic compound from solution. As discussed in the introduction, the ionic compound precipitated in this last step is a hydrate which includes 12 water molecules within each formula unit. The net ionic equation representing the formation of the solid product is



In full molecular form, this equation becomes:



Questions

The chemical equations for the 3 steps of this synthesis were presented above in full molecular form labeled 1, 2, and 3; and in net ionic form labeled 1i, 2i, and 3i.

Combine equations 1, 2, and 3 to find a full molecular equation for the synthesis of alum. Notice that you will need to include equations 2 and 3 multiplied by a factor of 2 to properly cancel the intermediate formed in step 1.

In a similar manner, combine equations 1i, 2i, and 3i to obtain a net ionic equation for the synthesis of alum. Again, the last two equations must be multiplied by a factor of 2.

Analysis

Use the full molecular equation for the synthesis along with the formula weight of alum determined above to calculate a theoretical yield of alum assuming that the aluminum metal is the limiting reactant.

From your data, find the net mass of alum produced. This is the actual yield.

Determine the percentage yield in the synthesis by comparing the actual yield to the theoretical yield.

In the discussion, summarize the entire procedure by including the full molecular equation for the entire synthesis and stating how each reactant was added and what happened to each product. (Example: "The KOH was added in step 1 as 25 mL of a 1.4 M solution to the aluminum metal which caused ...")

You should have obtained less than 100% yield. Your discussion should speculate on the fate of the rest of the potential product.