

Preparation of Common Alum

Objective: To perform a series of reactions to transform aluminum foil into common alum, $KAl(SO_4)_2 \cdot 12 H_2O$.

Concept to be Tested: It is possible to transform one compound into another by performing one or more reactions.

Text Reference: McMurray and Fay: Chapter 3.4-3.9 and Chapter 4.3-4.5

Techniques: Review *General Lab Procedures* (theoretical and percent yield calculations) and *Filtration* (vacuum and gravity filtration)

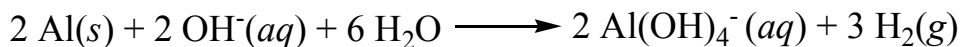
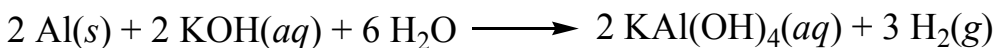
Introduction

Despite its name, alum doesn't necessarily have anything to do with aluminum, even though this experiment will be used to prepare common alum from aluminum foil. Alum, in chemistry, is a term given to the crystallized double sulfates of the typical formula $M_2SO_4 \cdot M^{III}(SO_4)_3 \cdot 24 H_2O$, in which M stands for an alkali metal (Li, Na, K, Rb, or Cs) and M^{III} is a trivalent ion most commonly Al^{3+} , Fe^{3+} , or Cr^{3+} (other trivalent ions associated with alums include gallium, vanadium, manganese, and cobalt). All alums are water soluble and have an astringent, acid, and sweet taste. They react as acids with litmus (turn blue litmus red). The simplified formula of alums is $MM^{III}(SO_4)_2 \cdot 12 H_2O$.

FYI. Pliny was the first to publish a reference of *alumen*, which we translate into alum. This material was well known to the ancients who frequently used their alum and green vitrol (iron sulfate) for medicinal purposes. Actually many soluble sulfates were lumped together under the name alumen (alum). The alumen used by the ancient Greeks and Romans was most probably the iron form of alum. There was a whitish version of their *alumen* found along lakes and on some rocks called *schistos alumen*. This schistos alum was most probably a mixture of aluminum and iron containing alums associated with some bituminous shales. The alumen of the ancients is most certainly not the alums known to the moderns. Even alchemists were a bit loose with their definition of alum. It wasn't until 1797 when Vauqueline distinguished what we call alums as being double salts that contained sulfate.

Alum is used today for hardening photographic film, to prepare pickles, to create dyes, to fix dyes (mordant), and to make lakes (insoluble dyes used in make up and food when the color absolutely must not run or bleed). Alum is also used in solutions used to soak sore joints and to reduce swelling. Shaving styptic (use to stop bleeding from nicks and cuts) is commonly potassium alum although straight aluminum sulfate is sometimes used.

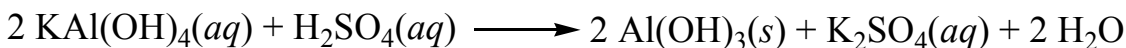
Following is a brief description of how you will prepare alum. Aluminum is a fairly reactive metal that will react (dissolve) in strong bases. The chemical reactions (balanced formula and net ionic equations) involved are shown below.



FYI. This is what happens when you add crystal Drano[®] to a clogged sink. The solid is either sodium or potassium hydroxide (whichever is cheaper at the time) and small chunks of aluminum metal. This reaction gets very hot (melts grease) and the hydrogen gas evolved causes the churning reaction that finally dislodges the plug.

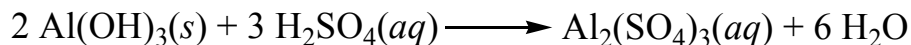
Aluminum oxide and aluminum hydroxide are amphoteric (meaning they react with both acids and bases). The aluminum hydroxide that would be formed by the reaction continues to react to form the soluble tetrahydroxoaluminate(III) ion, $[\text{Al}(\text{OH})_4]^-$. This is how the raw aluminum containing ores are purified. The ore is dissolved in strong base and the aluminum in the ore winds up as the soluble ion in solution that can then be filtered and purified.

Neutralization of this solution produces aluminum hydroxide. Sulfuric acid is shown in this reaction although any acid could be used.



FYI. In the commercial manufacture of aluminum metal, atmospheric CO_2 is bubbled through the solution to acidify it. The precipitate is filtered and then roasted to remove all water and produce alumina, Al_2O_3 , which is then shipped to a plant either in Pennsylvania or Texas where, following further reactions to produce the suitable salts, aluminum is isolated from by electrolysis of the molten salt. In all, the isolation of aluminum from its raw ores, is a labor and energy intensive process. The EPA estimates that it takes one-fourth the energy to produce aluminum from a recycled can or other aluminum containing product. Recycling rates have been dropping steadily in the US for the past 27 years.

The insoluble aluminum hydroxide is neutralized in today's experiment by excess sulfuric acid to form soluble aluminum sulfate.



Since this neutralization has been run in a single container, there are still K^+ ions present in the solution. When the solution cools, the double salt, $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$, hydrated potassium aluminum sulfate crystallizes out of solution. You will collect these crystals by filtration.

Experimental Procedure

This experiment uses some very corrosive materials. Eye protection must be worn at all times. Make certain to wash your hands before you leave the lab.

This experiment is very long so you must be prepared to start the experiment the moment you get into the laboratory. Your instructor may request that you bring a clean disposable plastic drink bottle with you to lab so that you may store your reaction mixture.

Obtain a piece of aluminum foil approximately 6" square, weigh this piece of foil (to 0.0001 g) and record the weight. Tear the foil into smaller pieces and put the pieces into a clean 250 mL beaker. **IMPORTANT! Make certain that the beaker is free of any cracks or stars.**

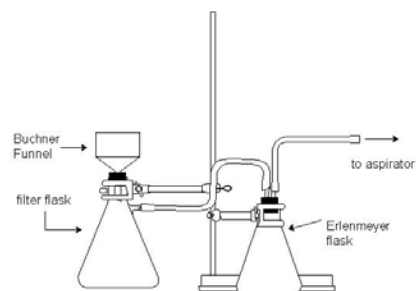
Once you have all of the foil in the beaker, add a magnetic stir bar. Make certain you can identify your beaker and move it to the hood (if at all possible). Now add ~30 mL of 4 M KOH to the beaker. **Make a mark (wax pencil, marker, or tape) to indicate the height of the liquid within the beaker (important for later).** Within a few minutes a vigorous reaction should begin. This reaction generally produces a mist that smells bad (and is somewhat corrosive as well). Within a few minutes (up to 5 min), the vigorous bubbling will subside. At this time, move the beaker back to your workstation and place it on a hot plate stirrer (**heat OFF**). You can now begin stirring the mixture. If any pieces of foil have migrated up the sides of the beaker you can push these back into the solution either with a glass stirring rod or use a squirt bottle of distilled water and wash these pieces back into the beaker with small quantities of water.

When no more small pieces of aluminum are visible in the stirred solution, carefully add 30 mL of 6 M H₂SO₄. (Do not worry if the solution is very dark, but do make certain all visible aluminum slivers are gone before you add the sulfuric acid.) Within moments a large quantity of cottony white precipitate will form. Now begin heating the reaction mixture. You will want to heat the mixture to boiling (make certain to get the mixture stirring early on). The solution should eventually go water clear. Now all you need to do is to reduce the volume by boiling. Allow the solution to boil until the liquid level is just above the line you made when you added the 30 mL of KOH. If the liquid volume drops below this line, you will need to add distilled water from the squirt bottle to the beaker to raise the level. Remove the beaker from the stirring motor using beaker tongs or a folded paper towel. (**HINT! The beaker is very hot.**) Place the beaker on your lab bench to begin cooling.

This is a convenient place to stop the reaction and store it for the next laboratory period. Store your mixture if your instructor directs you to do so. Make certain that you will be able to identify your mixture when you return to lab.

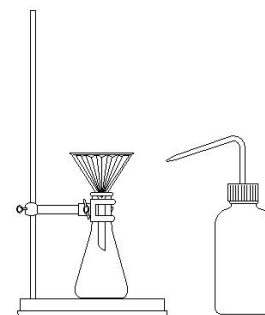
It is best to allow the mixture to cool slowly for best crystal formation. If the solution cools completely and no crystals appear, either scratch the side of the beaker with a stirring rod or add a small seed crystal to initiate crystallization. If neither of these procedures works, cool the solution in an ice bath. (Use of an ice bath will result in the formation of very small crystals.)

Vacuum Filtration. Set up a vacuum filtration, choose a piece of filter paper that just fits inside the Büchner funnel and covers the holes. Weigh the filter paper (to 0.0001 g) and record the weight. Turn on the water full force; squirt a small amount of distilled water onto the filter paper so that it will stay flat on the surface of the filter. Now pour the cooled solution into the filter and allow the liquid to drain away. Rinse any crystals out of the container and onto the filter by using a small quantity of alcohol or acetone (~20 mL). *Do not* use water because your product is water-soluble.



When the suction has pulled the filter to near dryness, break the suction (then turn off the water), and carefully remove the filter paper and crystals. [Dispose of the liquid in a jug provided for this purpose in the hood.] Place the filter paper and crystal onto a large watch glass and place this into the oven to dry. (**Important!** *The oven temperature cannot exceed 80 °C – alum melts below 100 °C.*) When the paper and crystals are dry (10 – 20 min), remove the watch glass from the oven, allow it to cool and then weigh the filter paper and crystals. You may discard the alum and filter paper in the wastebasket. Use the data you collect to complete your lab report and calculate the percent yield of alum.

Gravity Filtration. You may be required to do a gravity filtration of your product rather than a vacuum filtration. You will need to clamp your flask to a ring stand. Obtain and weigh a piece of filter paper (record this weight). Fold the filter paper (either folded in quarters and opened, or fluted) and place it into the filter. Use a *small* quantity of water to dampen the filter paper to hold it down. Since this will be time consuming, try to decant (pour off) at least half of the liquid into the filter (if possible) before allowing



crystals into the filter. This should allow the fastest possible draining. Then pour the rest of the contents into the filter. You will need to wash any remaining crystals from the container (beaker or bottle) using a squirt bottle of acetone or water. When filtered, use a small quantity of acetone or alcohol to wash the filter and your product. [Dispose of the liquid in a jug provided for this purpose in the hood.] Place the filter paper and crystal onto a large watch glass and place this into the oven to dry. (**Important!** *The oven temperature cannot exceed 80 °C – alum melts below 100 °C.*) When the paper and crystals are dry (10 – 20 min), remove the watch glass from the oven, allow it to cool and then weigh the filter paper and crystals. You may discard the alum and filter paper in the wastebasket. Use the data you collect to complete your lab report and calculate the percent yield of alum.

Your instructor may choose to have you store your crystals until the next lab period so that they may air dry rather than subject them to oven drying. If this is the case, make certain your crystals are tightly wrapped in the filter and this placed securely with paper towels. You will need to make certain that you can identify your crystals when you return to lab.

Cleanup

Wash and dry your glassware and stirring bar then return the items to their proper storage locations. Should your beaker contain a white residue that you cannot remove, ask your Instructor for a small amount of HCl. The residue is aluminum hydroxide and will readily dissolve in dilute HCl. (Dispose of this in the waste bottle in the hood.) Wrap the cord neatly around the hot plate and then carefully stack it in the hot plate storage area. If a plastic bottle was used to store your product between laboratory periods, it may be rinsed with water and safely discarded in the wastebasket.

Safety

You must wear eye protection at all times during this laboratory experiment. Both the KOH and H₂SO₄ solutions are corrosive and hazardous. The reaction tends to produce an aerosol and also tends to splatter. Wipe up any spills immediately with paper towels and rinse the area with water. Should you get either material on yourself, wash the area with copious quantities of water. Wash your hands *before* you leave the laboratory. Dispose of the liquids you collect in the bottle in the hood labeled for this purpose. Your product and filter paper may be safely disposed of in the wastebasket.

