

Synthesis of Alum, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$

In this experiment the ionic compound, potassium aluminum sulfate ($\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$), will be prepared from a water solution that contains K^+ , Al^{3+} and SO_4^{2-} (potassium, aluminum, and sulfate ions, respectively). The aluminum ions will be formed by oxidizing aluminum from aluminum foil. The "double salt" potassium aluminum sulfate dodecahydrate is commonly referred to as alum. Many combinations of mono- and tri-positive cations yield crystals of the same stoichiometry and structure, and alum is a general name for this type of compound. For example, there is chrome alum, $\text{KCr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, which is a deep purple color, as well as alums where either sodium or ammonium ions are present instead of the potassium ion. The crystals are usually in the form of octahedra.

If an aqueous solution which contains $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$ ions, K^+ ions and SO_4^{2-} ions is allowed to evaporate, the compound $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ will crystallize. Within the alum crystal, six waters of hydration are bonded directly to the aluminum ion to give $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$ ions, while the other six surround the K^+ ion.

Alum crystals of great purity are easily prepared. Because of this purity, alum is useful in the dyeing of cloth, where the alum acts as a source of Al^{3+} ions which are not contaminated with Fe^{3+} . The Al^{3+} is precipitated on the cloth as aluminum hydroxide which acts as a binding agent for dyes. It is necessary that no Fe^{3+} be present in order to produce clear colors.

Aluminum is considered a reactive metal, but because its surface is usually protected by a thin film of aluminum oxide, it reacts only slowly with acids. It does, however, dissolve quickly in basic solutions. Excess hydroxide ion converts the aluminum to the tetrahydroxoaluminate(III) ion, $[\text{Al}(\text{OH})_4]^-$. When acid is slowly added to this ion, white, gelatinous aluminum hydroxide ($\text{Al}(\text{OH})_3$) precipitates. Continued addition of acid causes the hydroxide ions to be completely neutralized, and the aluminum exists in solution as the hydrated ion $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$. Aluminum hydroxide is considered to be an "amphoteric" hydroxide because it dissolves in both acids and bases.

Chemicals

Aluminum foil	Potassium hydroxide, KOH, 3 M
Sulfuric acid, H_2SO_4 , 3 M	Water-ethanol solution, 50% by volume
Baking soda, $\text{NaHCO}_3(\text{s})$	Vinegar, dilute $\text{HC}_2\text{H}_3\text{O}_2$

Equipment

Beaker, 250 mL	Watch glass
Graduated cylinder	Stirring rod
Büchner funnel and filter flask	Burner, ring stand, ring, wire gauze
Ice bath	Plastic wrap or Parafilm®
Balance	Fume hood

Procedure

1. Weigh Out and Dissolve the Aluminum.

Weigh out about 1 gram of aluminum foil to the nearest centigram. Tear the foil into small pieces and place in a 250 mL beaker.

Safety Alert

You will be using solutions with high concentrations of sulfuric acid and potassium hydroxide, both of which are highly damaging to skin and eyes. Be careful when handling them. If you spill any on yourself, wash off with lots of water. Neutralize sulfuric acid spills on the counter with baking soda, and neutralize potassium hydroxide spills with vinegar (dilute acetic acid).

When aluminum dissolves in potassium hydroxide solution, hydrogen gas is produced. Make sure that no flames are present. This step should be performed in a fume hood.

Wear Chemical Splash Goggles and a Chemical-Resistant Apron.

Slowly add 25 mL of 3 M KOH solution. Allow the reaction to proceed until all of the foil is dissolved. Remove any undissolved solids such as carbon particles by filtering the solution through a Büchner funnel while the solution is hot. Rinse the filter paper with a small amount of distilled water.

2. Acidify with Sulfuric Acid.

At this point the solution contains $[\text{Al}(\text{OH})_4]^-$ and K^+ ions, along with excess OH^- ions. Cool the solution and then acidify it SLOWLY, with constant stirring, using 35 mL of 3 M H_2SO_4 . The solution will get very hot because you are adding strong acid to the strongly basic solution. Solid $\text{Al}(\text{OH})_3$ will first precipitate and then dissolve as more H_2SO_4 is added. If a precipitate still remains, filter the solution and discard the solids. You can use vacuum filtration with a Büchner funnel and filter flask to speed up this process. Then boil the solution until water has evaporated to give a volume of about 50 mL of solution. This is a good place to stop if the end of the lab period is near. Cool the solution and cover the beaker with Parafilm® or plastic wrap. Allow it to rest undisturbed until the next period.

3. Crystallize the Alum.

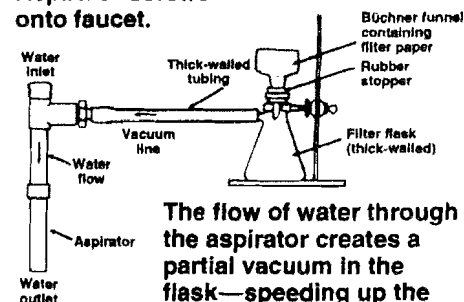
If time permits, cool the solution in an ice bath for 15 minutes, keeping it as motionless as possible. Crystals of alum should grow in the beaker. If no crystals form, scrape the bottom of the beaker with a stirring rod to create a rough place where crystals may begin to grow, or add a seed crystal. If there are still no crystals, reheat the solution until more water has evaporated and then cool again. Rapid cooling in an ice bath causes very small crystals to grow; slow overnight cooling allows the formation of larger crystals. Collect the alum crystals by vacuum filtration. Wash the crystals with 50 mL of a 50% by volume water and ethanol mixture, in which alum crystals are not very soluble. Allow the crystals to dry at room temperature. Determine the mass of the alum. Calculate the theoretical yield of alum assuming that aluminum was the limiting reactant and that the foil was 100% aluminum, and calculate your percent yield. (SAVE IN VYLE)

Verify that your crystals are alum by performing Experiment 4, "Analysis of Alum."

HOW TO...

SET UP A VACUUM FILTRATION

Aspirator screws onto faucet.



The flow of water through the aspirator creates a partial vacuum in the flask—speeding up the filtration process.

EXPERIMENT THREE

Disposal

Pour the filtrate into a beaker which your instructor will provide for disposal. See the *Flinn Chemical Catalog/Reference Manual*, suggested disposal method #24b. See the appendix.

Discussion

Answer the following questions in your laboratory report:

1. Write balanced net ionic equations for the following reactions which occur in this synthesis:
 - a) Aluminum reacts with KOH and water forming potassium ions, $[\text{Al}(\text{OH})_4]^-$ and hydrogen gas.
 - b) Hydrogen ions from the acid react with the tetrahydroxoaluminate ions to precipitate aluminum hydroxide.
 - c) Aluminum hydroxide reacts with additional hydrogen ions and water to form $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$
 - d) Alum forms from the potassium ions, hydrated aluminum ions, sulfate ions and water.
2. What is a "synthesis" reaction?
3. Why should you NOT expect a 100% yield of crystals?
4. How does the solubility of alum in water change with temperature?
5. Why should you NOT wash the crystals with pure water?
6. What do your crystals look like?
7. What is the shape of an octahedron?
8. Show how you obtained your theoretical and percent yields.

mass of alum

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Preliminary Lab Assignment

Name _____ Date _____ Class _____

1. What is alum?
2. What is a hydrated crystal?
3. What is meant by the term amphoteric?
4. Write a balanced net ionic equation showing aluminum hydroxide dissolving in a solution containing excess hydroxide ion.
5. Write a balanced net ionic equation showing aluminum hydroxide dissolving in an acid solution.
6. If one carried out a reaction to synthesize $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ using 1.0 g of potassium metal as the limiting reagent, what would be the maximum mass of alum that could form? Show your calculations.
7. If one carried out the procedure in question 6 and actually obtained 4.5 g of alum, what would be the percent yield? Show your calculations.