Lab 04.5
Synthesis of Alum

AP Chemistry

Alums are ionic compounds that crystallize from solutions containing sulfate ion, a trivalent cation such as Al^{3+}, Cr^{3+}, or Fe^{3+} and a monovalent cation such as K^+, Na^+, or NH_4^+. Six of the water molecules bind tightly to the trivalent metal ion; the remaining six molecules bind more loosely to the monovalent cation and the sulfate anion.

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 $A1^{3+}$ ions which are not contaminated with Fe^{3+}. The $A1^{3+}$ 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 to produce clear colors.

The synthesis of alum, $KAl(SO_4)_2 \bullet 12H_2O$, can be accomplished through the following reactions. Aluminum is first oxidized by potassium hydroxide to form a soluble salt in the chemical reaction.

$$2\ Al(s) + 2\ KOH(aq) + 6\ H_2O(l) \rightarrow 2\ KAl(OH)_4(aq) + 3\ H_2(g)$$

After filtration to remove any residual plastic or paint, the solution is clear and colorless. The H_2 is evolved as a gas and mixes with the atmosphere. The chemical species in solution are K^+, $Al(OH)_4^-$, and unreacted KOH. Sulfuric acid is added and two sequential reactions occur. Initially, as the acid is added, a precipitate, $Al(OH)_3$ forms.

$$2\ KAl(OH)_4(aq) + H_2SO_4(aq) \rightarrow 2\ Al(OH)_3(s) + 2\ H_2O(l) + K_2SO_4(aq)$$

Then as more sulfuric acid is added the precipitate dissolves

$$2\ Al(OH)_3(s) + 3\ H_2SO_4(aq) \rightarrow Al_2(SO_4)_3(aq) + 6\ H_2O(l)$$

to give aluminum ions, Al^{3+}, in solution. The solution at this point contains Al^{3+}, K^+ and SO_4^{2-} ions. On cooling these ions form a solid hydrate, $KAl(SO_4)_2 \bullet 12\ H_2O$ (Alum).

$$Al_2(SO_4)_3(aq) + K_2SO_4(aq) + 12\ H_2O(l) \overset{\text{cold}}{\rightarrow} 2\ KAl(SO_4)_2 \bullet 12\ H_2O(s)$$

In the experiment the crystallization can be sped up by providing a small seed crystal of alum for new crystals to form upon. Cooling is needed because alum is soluble in room temperature water. Finally, the crystals of alum are removed from the solution by vacuum filtration and washed with an alcohol/water mixture. This wash liquid removes any contamination from the crystals and dries quickly because alcohol is relatively volatile.

Safety

- The concentrated sulfuric acid and potassium hydroxide used in this lab are both highly damaging to skin and eyes. Splash goggle are required and aprons are highly recommended. Spills should be neutralized and cleaned up immediately. Any solutions that contact skin should be rinsed off with plenty of water.
- During the dissolution of the aluminum foil, hydrogen gas is produced. This step should be performed in a fume hood and no flames or heat sources should present in the area.

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I. PURPOSE

To learn how to make and record valid laboratory observations.

II. MATERIALS

1. Aluminum foil
2. 3*M* H_2SO_4 *(aq)*
3. 3*M* KOH *(aq)*
4. 50% ethanol solution
5. pH paper
6. 250 mL beaker
7. Graduated cylinder
8. Filter funnel
9. Filter paper
10. Büchner funnel
11. Filter flask
12. Analytical balance
13. Watch glass
14. Stirring rod
15. Parafilm
16. Ring stand & ring
17. Bunsen burner
18. Wire gauze

III. PROCEDURES

Day 1

1. Clean all glassware.
2. Obtain about 1 gram of aluminum foil. Record the mass of the aluminum to ± 0.001 gram. Tear the aluminum into small pieces and place in a clean 250 mL beaker. *(Do not lose any of the foil.)*
3. In the fume hood, slowly add 25 mL of 3*M* KOH to the beaker containing the aluminum. *(This step will produce fumes and heat, so proceed slowly and carefully.)* Stir the combination with a clean stirring rod until the reaction is complete.
4. Place a clean funnel, with a piece of folded filter paper, on a 125 mL Erlenmeyer flask. Remove any undissolved solids by gravity filtering the solution while the solution is still hot. *Only fill the funnel to within ½" of the top of the paper.* Use a glass rod to "guide" the solution into the paper (as demonstrated by your instructor). The solution in the Erlenmeyer flask should be both clear and colorless at this point. Dispose of the solids and filter paper.
5. Allow the flask to cool. While it is cooling, wash the funnel and beaker with lots of tap water to remove any potassium hydroxide.
6. Add 35 mL of *3M* H_2SO_4 to the flask. *Swirl the flask as you add the acid.* The solution will get very hot because you are adding strong acid to the strongly basic solution. Solid will first precipitate and then dissolve as more H_2SO_4 is added. If there are any white flecks left in the solution after the addition of the H_2SO_4, place the flask on the Bunsen burner apparatus and warm it with swirling until all of the solid material has dissolved.
7. Allow the flask to cool a little and then place it in the ice bath. Allow it to cool for an additional 5 minutes.
8. If alum crystals have not started to form, scratch the inside walls of the flask with a stirring rod. This provides sites at which crystallization can begin, followed by crystal formation throughout the liquid. Swirl the flask when you notice the onset of crystal formation and allow it to cool in the ice bath for another 10 minutes.
9. While the solution is cooling, pour 20 mL of 50% alcohol/water mixture into a test tube and place it in the ice bath to cool.
10. Weigh a piece of filter paper and record its mass.
11. Prepare a vacuum filtration apparatus as illustrated by your instructor. Wet the filter paper with distilled water.
12. Remove the flask containing the alum crystals from the ice bath, swirl so that all the crystals are dislodged, and pour quickly into the Buchner funnel. Keep swirling and pouring until all the solution and crystals are transferred to the funnel.
13. Pour about 10 mL of the cooled alcohol/water mixture into the flask. Swirl the flask and pour mixture into the funnel to transfer any remaining crystals.
14. Label a clean dry watch glass with your period and group number. Carefully remove the filter paper and crystals from the funnel and place on the watch glass. Set aside in a place designated by your instructor for drying overnight.
15. Pour the filtrate into a beaker which your instructor will provide for disposal and clean the filtering flask for storage.

Day 2

16. Record the mass of the dried filter paper and alum.
17. Store your crystals as directed by your instructor for use in a later lab.

IV. PRE-LAB QUESTIONS

1. What is a "synthesis" reaction?
2. What is a hydrated crystal?
3. Why must Step #2 be completed in the fume hood?
4. Why should you NOT wash the crystals with pure water?

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V. DATA & CALCULATIONS

A. DATA

Table A – Data

Mass of aluminum foil (g):	
Volume of *3M* KOH *(aq)* :	
Volume of *3M* H_2SO_4 *(aq)*:	
Mass of filter paper (g):	
Mass of filter paper + alum (g):	
Mass of alum produced (g):	

B. CALCULATIONS

1. Calculate the theoretical yield of alum assuming aluminum was the limiting reactant and that the foil was 100% aluminum.
2. Calculate your percent yield.

VI. QUESTIONS & DISCUSSION OF ERROR

A. QUESTIONS

1. Describe the solution present at the end of Step #9. How does this affect the percent yield?
2. How does temperature affect the solubility of alum in water? Cite evidence from your lab to support your answer.
3. Describe the appearance of the crystals produced. Is there a geometric pattern?
4. Write balanced net ionic equations for each equation given in the lab background section.

B. DISCUSSION OF ERROR

VII. CONCLUSION