Name: ____ Section: ______________

Experiment 4: Synthesis of Alum Pre-laboratory Assignment
(Read through the experiment before starting!)

1. a) What are the strong acid and strong base used in this synthesis?

b) What should you do if you spill the acid or base on your skin?

2. In your synthetic procedure, you use 0.6343 g aluminum metal, 50.3 mL of 1.43 M KOH, and 20.2 mL of 9.2 M H_2SO_4. a) Which reactant is the limiting reactant? Show your work for credit.

b) What is your theoretical yield in grams?

EXPERIMENT 4: SYNTHESIS OF AN ALUM

Introduction

Chemical synthesis refers to the process of making new substances from other substances. Alums are ionic compounds that are made up of a monovalent cation (e.g., K^+, Na^+, NH_4^+), a trivalent cation (e.g., Al^{3+}, Cr^{3+}, Fe^{3+}), two sulfate anions (SO_4^{2-}), and twelve water molecules. In this experiment we will synthesize aluminum potassium sulfate dodecahydrate ($KAl(SO_4)_2 \bullet 12\ H_2O$) from aluminum foil and other basic chemicals. This alum is widely used to dye fabrics, make pickles crunchy, and manufacture paper. It is also used as a coagulant in water purification and as an astringent in medicine. This alum, like all alums, is a *hydrate* meaning that it is comprised of a salt and water combined in definite proportions. The dot between $KAl(SO_4)_2$ and $12\ H_2O$ in the formula indicates that twelve moles of water are associated with every mole of $KAl(SO_4)_2$. Therefore, the mass of the twelve water molecules, called the *waters of hydration,* must be included in the molecular weight of the compound.

$KAl(SO_4)_2 \bullet 12\ H_2O$ is prepared from aluminum (Al), potassium hydroxide (KOH), and sulfuric acid (H_2SO_4). The four reactions involved in this synthesis are outlined below.

(1) $2\ Al_{(s)} + 2\ KOH_{(aq)} + 6\ H_2O_{(l)} \rightarrow 2\ K[Al(OH)_4]_{(aq)} + 3\ H_{2(g)}$

(2) $2\ K[Al(OH)_4]_{(aq)} + H_2SO_{4(aq)} \rightarrow 2\ Al(OH)_{3(s)} + K_2SO_{4(aq)} + 2\ H_2O_{(l)}$

(3) $2\ Al(OH)_{3(s)} + 3\ H_2SO_{4(aq)} \rightarrow 2\ Al^{3+}{}_{(aq)} + 3\ SO_4^{2-}{}_{(aq)} + 6\ H_2O_{(l)}$

(4) $K^+{}_{(aq)} + Al^{3+}{}_{(aq)} + 2\ SO_4^{2-}{}_{(aq)} + 12\ H_2O_{(l)} \rightarrow KAl(SO_4)_2 \bullet 12\ H_2O\ (s)$

Notice in the reaction sequence, equation 4 must be multiplied by a factor of two for it to contain the same moles of aluminum as equations 1 thru 3. In equations 5 thru 8, the original four equations have been rewritten as ionic equations with equation 4 multiplied by two.

(5) $2\ Al_{(s)} + 2\ K^+{}_{(aq)} + 2\ OH^-{}_{(aq)} + 6\ H_2O_{(l)} \rightarrow 2\ K^+{}_{(aq)} + 2\ [Al(OH)_4]^-{}_{(aq)} + 3\ H_{2(g)}$

(6) $2\ K^+{}_{(aq)} + 2\ [Al(OH)_4]^-{}_{(aq)} + 2\ H^+(aq) + SO_4^{2-}{}_{(aq)} \rightarrow$
$2\ Al(OH)_{3(s)} + 2\ K^+{}_{(aq)} + 2\ SO_4^{2-}{}_{(aq)} + 2\ H_2O_{(l)}$

(7) $2\ Al(OH)_{3(s)} + 3\ H_2SO_{4(aq)} \rightarrow 2\ Al^{3+}{}_{(aq)} + 3\ SO_4^{2-}{}_{(aq)} + 6\ H_2O_{(l)}$

(8) $2\ K^+{}_{(aq)} + 2\ Al^{3+}{}_{(aq)} + 4\ SO_4^{2-}{}_{(aq)} + 24\ H_2O_{(l)} \rightarrow 2\ KAl(SO_4)_2 \bullet 12\ H_2O\ (s)$

Equation 9 results when we sum equations 5 thru 8, cancel identical reagents on both sides of the equations, and pair ions according to the reagents used and products isolated. Equation 9, the overall equation for the synthesis of alum, will be useful in determining the limiting reagent.

(9) $2\ Al_{(s)} + 2\ KOH_{\ (aq)} + 4\ H_2SO_{4\ (aq)} + 22\ H_2O_{(1)} \rightarrow 2\ KAl(SO_4)_2 \bullet 12\ H_2O_{(s)} + 3\ H_{2(g)}$

Objectives

In this experiment, you will (1) prepare $KAl(SO_4)_2 \bullet 12\ H_2O$ from aluminum foil, (2) determine the limiting reagent in the synthesis, and (3) calculate the theoretical and percent yield of product.

Safety Precautions

In this experiment, you will be working with sulfuric acid (H_2SO_4), a strong acid, and potassium hydroxide (KOH), a strong base. In case of contact with either, wash the affected area immediately with a large amount of water and notify your instructor. Also, hydrogen gas, which is highly flammable, is generated in the first reaction of the experiment, so avoid open flame.

Experimental Procedure

1. *Synthesis of Alum*

a. Weigh approximately 0.5 g of aluminum foil to the nearest 0.001 g and record the value.

b. Place the aluminum in a 250 mL beaker and add 50 mL (record the volume to the nearest 0.1 mL) of 1.4 M potassium hydroxide (KOH). Bubbles of hydrogen gas should form within a few seconds. Carefully place a watchglass over the beaker. To speed up the reaction, heat the beaker gently on a hot plate. If the liquid level in the beaker drops below 25 mL, add enough deionized water to bring the liquid level to 30-35 mL. You may have to add water more than once during the reaction to maintain the liquid level at 30-35 mL. The reaction is complete when no more bubbles of hydrogen gas form. The reaction is usually complete in about twenty minutes. While you are waiting for the reaction to reach completion, assemble a Buchner filtration apparatus as in Figure 1. Be sure to clamp the filtration flask to a ring stand. Place a piece of filter paper inside the Buchner funnel and moisten it with a little deionized water. Also, prepare an ice bath by halfway filling a 600 or 800 mL beaker with crushed ice and then covering the ice with cold water.

c. When the reaction is complete, filter the mixture through the Buchner funnel by turning on the faucet to begin suction and then pouring the mixture into the Buchner funnel. Once all of the liquid has passed through the funnel, turn off the suction. Transfer the solution in the filter flask (called the filtrate) to a 250 mL beaker and cool the solution to room temperature in an ice bath. The gray solid and filter paper in the funnel should be disposed of in the appropriate waste container.

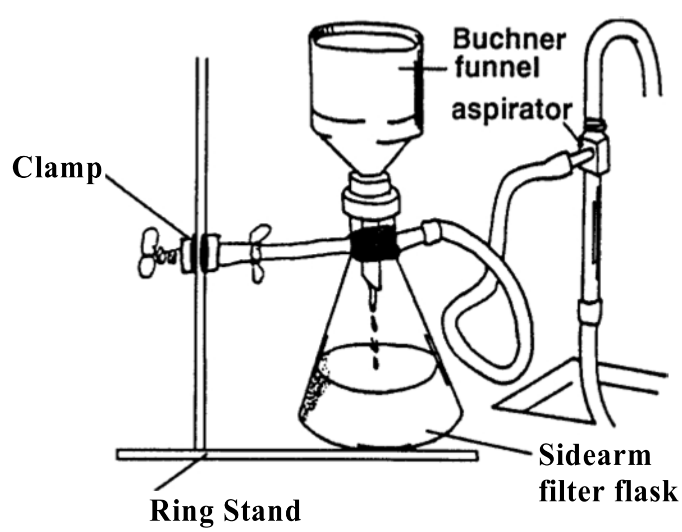

Figure 1.

d. Remove the room temperature solution from the ice bath. While continuously stirring the solution, slowly add 20 mL (record the volume to the nearest 0.1 mL) of 9.0 M sulfuric acid (H_2SO_4). Large clumps of fluffy white aluminum hydroxide $Al(OH)_3$ should form. Gently heat and stir the mixture for 10 minutes to dissolve the $Al(OH)_3$. If any solid remains after heating, filter the mixture. The total solution volume should **not exceed** 40 mL. If you have more than 40 mL of solution, boil the solution until the volume is less than 40 mL.

e. Chill the solution in an ice bath for about 15 minutes. Alum crystals should form. It may be necessary to scratch the side of the beaker gently with a glass rod to induce crystallization. Because alum is less soluble in water than other byproducts formed in this experiment, the byproducts will mostly remain in solution. While the alum is crystallizing, chill 10 mL of a 50/50 (v/v) ethanol-water solution in your ice bath.

f. Filter the slurry through the Buchner funnel as in step 3 and be sure to swirl the mixture before pouring it into the funnel. Add the 10 mL of cold 50/50 ethanol-water solution to the crystals remaining in the 250 mL beaker, swirl the mixture, and, with the aspirator still on, pour the mixture into the Buchner funnel. Use a rubber policeman to transfer any crystals remaining in the 250 mL beaker to the funnel. Rinse the crystals in the Buchner funnel with 5 mL of 95% ethanol. Dry the crystals by aspirating (drawing air through the cake of crystals in the funnel by maintaining suction) for ten minutes. Turn off the aspirator.

g. Use a rubber policeman to carefully transfer the alum crystals from the Buchner funnel to a clean, dry, **pre-weighed** watchglass. Carefully place the watchglass with the crystals in your drawer and allow them to dry until the next lab period. During the next lab period, weigh the alum to the nearest 0.001 g.

Calculations

In this experiment, you are asked to calculate the *theoretical yield,* or the maximum amount of alum that can be synthesized from 0.500 g of alum, 50.0 mL of KOH, and 20.0 mL of H_2SO_4. To do this, you must first determine which reactant is the *limiting reactant,* the reactant that is completely consumed in the reaction, and thus determines the maximum amount of product that can be made. To illustrate, consider the reaction of 6.0 g of hydrogen with 64.0 g of oxygen to produce water according to the following equation:

$$2\ H_{2(g)} + O_{2(g)} \rightarrow 2\ H_2O_{(l)}$$

First, convert the masses of hydrogen and oxygen to moles using their respective molecular weights. This yields 3.0 moles of H_2 and 2.0 mol of O_2. The equation's stoichiometric coefficients indicate that 3.0 moles of hydrogen will yield 3.0 moles of water because the hydrogen and water are in a 2:2 (or 1:1) ratio:

$$3.0\ \text{mol}\ H_2 \times \frac{2\ \text{mol}\ H_2O}{2\ \text{mol}\ H_2} = 3.0\ \text{mol}\ H_2O$$

Similarly, 2.0 moles of oxygen will yield 4.0 moles of water because the oxygen and water are in a 2:1 ratio:

$$2.0\ \text{mol}\ O_2 \times \frac{2\ \text{mol}\ H_2O}{1\ \text{mol}\ O_2} = 4.0\ \text{mol}\ H_2O$$

The H_2 is used up before the O_2 and limits the amount of H_2O that can be produced to 3.0 moles. Therefore, hydrogen is the limiting reagent and oxygen is in excess.

The amount of product that can be made when all of the limiting reagent reacts is the *theoretical yield.* The theoretical yield of H_2O in the example above is 3.0 moles or, converting moles to grams using the molecular weight of water, 54 g of water.

The amount of product that is actually obtained is known as the *actual* or *experimental yield.* The experimental yield is almost always less than the theoretical yield. *A percent yield,* which compares the experimental yield to the theoretical yield, is used to judge the efficiency of a reaction and the techniques used to synthesize a compound. If 50 g of water were actually produced in this example, the percent yield is 93%.

$$\% \text{ yield} = \frac{\text{Expt. yield (g)}}{\text{Theor. yield (g)}} \times 100 = \frac{50\text{ g}}{54\text{ g}} \times 100 = 93\,\%$$

At this point, you should be able to complete your lab report without reading the remaining portion of the calculations section. Please attempt to so and only consult the following example calculations if you become stumped. Following is a step-by-step set of suggestions detailing how to calculate the percent yield of alum.

1. Determine the moles of Al using your experimental mass of Al and the molecular weight of Al.

2. The molarity of the KOH you recorded should be from the reagent bottle label and should be close to the 1.4 M noted in part b of the experimental procedure. Calculate the moles of KOH from the molarity of KOH you recorded and the volume of KOH you used. Be sure to watch units, including mL versus L.

3. The molarity of the H_2SO_4 you recorded should be from the reagent bottle label and should be close to the 9.0 M noted in part d of the experimental procedure. Calculate the moles of H_2SO_4 from the molarity of H_2SO_4 you recorded and the volume of H_2SO_4 you used.

4. The moles of alum recovered are calculated form the mass of alum you recovered and the molecular weight of alum. Be sure to include the 12 waters of hydration in your calculation of the molecular weight of alum.

5. To determine the limiting reagent, calculate the theoretical moles of alum that could form from each of the three key reagents, Al, KOH, and H_2SO_4. We need not worry about the water because the reaction was conducted in aqueous solution, so we know water was in excess and was not the limiting reagent.

    a. For Al, the moles of Al in step 1 must be multiplied by the stoichiometric ratio of aluminum to alum in equation 9. The ratio is two moles of alum is produced for each two moles of Al used, so the values will be equal.
    b. For KOH, the moles of KOH in step 2 must be multiplied by the stoichiometric ratio of potassium hydroxide to alum in equation 9. The ratio is two moles of alum is produced for each two moles of KOH used, so, again, the values will be equal.
    c. For H_2SO_4, the moles of H_2SO_4 in step 3 must be multiplied by the stoichiometric ratio of aluminum to alum in equation 9. The ratio is two moles of alum is produced for each four moles of H_2SO_4 used. Notice here the theoretical moles of alum produced will be half that of the moles of H_2SO_4 used.

    Next, compare the three different "theoretical" moles of alum produced which you have calculated in steps a, b, and c. The smallest value is the true theoretical number of moles of alum. (You will need this value in step 6.) The reagent used to calculate the true theoretical moles of alum is the limiting reagent.

6. The theoretical yield of alum is simply calculated by multiplying the true number of moles of alum calculated in step 5 by the molecular weight of alum.

7. The percent yield of alum is calculated as described earlier:

$$\textbf{\%}\ \text{yield} = \frac{\text{Expt. yield (g)}}{\text{Theor. yield (g)}} \text{ x } 100$$

In today's synthesis:

$$\textbf{\%}\ \text{yield} = \frac{\text{mass of alum you isolated (step 4)}}{\text{Theor. yield of alum you calculated (step 6)}} \text{ x } 100$$

Experiment 4: Synthesis of Alum Data Sheet

Name: ______________________________
Section: _______

Data:

Mass of aluminum used ______________________________

Volume of KOH ______________________________

Molarity of KOH (from bottle) ______________________________

Volume of H_2SO_4 used ______________________________

Molarity of H_2SO_4 (from bottle) ______________________________

Mass of watchglass ______________________________

Mass of watchglass + alum ______________________________

Mass alum recovered ______________________________

Results and Calculations:

Moles of aluminum used ______________________________

Theoretical moles alum possible from aluminum used ______________________________

Moles of KOH used ______________________________

Theoretical moles alum possible from KOH used ______________________________

Moles of H_2SO_4 used ______________________________

Theoretical moles alum possible from H_2SO_4 used ______________________________

Limiting reactant ______________________________

Theoretical yield in grams ______________________________

Actual yield in grams ______________________________

Percent yield ______________________________

Instructor Date and Initial: ___________________________

Experiment 4: Synthesis of Alum Data Sheet **Name: ____________________________**

Section: _______

For full credit on this lab, you must show your work **CLEARLY** on this page: