

Name \_\_\_\_\_ Lab Day \_\_\_\_\_ Lab Time \_\_\_\_\_

## Experiment 3 · Synthesis of alum

### Pre-lab questions

Answer these questions and hand them to the TF before beginning work.

(1) What is the purpose of this experiment?

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(2) What is the formula of the alum you will prepare? What is its molar mass in grams per mole?

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(3) You will react Al(s) with 1.4 M KOH(aq): how will you know when the reaction is complete?

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(4) Immediately after the reaction of Al(s) with 1.4 M KOH(aq), you will filter the reaction mixture. Where is your product at the end of this filtration, in the filter flask or trapped on the filter paper?

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(5) Alum crystals should form after 9 M H<sub>2</sub>SO<sub>4</sub>(aq) is added to the cooled reaction mixture: what should you do if crystals do not form?

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# Synthesis of alum

Containers made from aluminum ( $\text{Al(s)}$ ) are quite durable: the average aluminum can lasts about 100 years. Before the enactment of laws requiring deposit on beverage containers, littering from discarded aluminum cans was a serious and costly problem. A more pressing issue is the wasteful depletion of aluminum reserves occasioned by the fabrication of single-use containers. Although aluminum is the most abundant metal in the Earth's crust, the supply of useful aluminum ores is limited and manufacturing the metal from its ores requires large amounts of electricity. Thus, recycling aluminum is a good idea.

In this experiment you undertake one of the most common ways in which aluminum is recycled: its conversion to potassium aluminum sulfate dodecahydrate ( $\text{KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O(s)}$ ), which is nicknamed "potassium aluminum alum" or simply "alum". Alum plays a role in a wide variety of applications from water purification, to leather tanning and dyeing, to baking and even in making pickles!

Whether used to make a soda can or to construct a Boeing 747 the surface of a sheet of aluminum is normally protected by a thin layer of aluminum oxide ( $\text{Al}_2\text{O}_3\text{(s)}$ ). This turns out to be a very fortunate happenstance: pure aluminum combines explosively with air. Thus, chemical and physical processes that compromise the integrity of the  $\text{Al}_2\text{O}_3\text{(s)}$  layer can be risky. This fact is most tragically demonstrated when an airliner crashes. The initial impact is usually sufficiently violent to rupture the gas tanks and ignite the fuel. The high temperature of the ensuing fire damages the  $\text{Al}_2\text{O}_3\text{(s)}$  layer covering the

airplane's fuselage and exposes the underlying aluminum to air, which then bursts into flame. You will remove the  $\text{Al}_2\text{O}_3(\text{s})$  layer protecting a piece of aluminum; luckily, the aluminum will be under water while you do so.

## Procedure

Bring an empty aluminum beverage can to lab. You should know how to calculate molar masses, how to perform stoichiometric calculations and how to determine percent yields. Consulting Appendix B "Stoichiometric Calculations" of this lab manual might help in answering the post-lab questions.

### Preparation of the aluminum sample

Cut from your can a piece of aluminum ( $\text{Al}(\text{s})$ ) measuring approximately  $5 \text{ cm} \times 7.5 \text{ cm}$  (about the size of a playing card). Use steel wool to scrape off as much paint as possible. Weigh the cleaned sample to two decimal places; you need about 1 g of  $\text{Al}(\text{s})$ . Record the mass of the sample in your notebook. Cut the aluminum into small squares measuring about 0.5 cm on edge. Smaller squares result in a faster rate of reaction.

### Reaction of aluminum with aqueous potassium hydroxide

The first step in the synthesis of alum is the reaction of aluminum with excess aqueous potassium hydroxide ( $\text{KOH}(\text{aq})$ ). Conduct the following operations in a hood.

Place the  $\text{Al}(\text{s})$  clippings in a 250-mL beaker and add 50 mL of 1.4 M  $\text{KOH}(\text{aq})$  solution. Cover the beaker with a clean watchglass (The set-up should not look like a dome: position the watchglass the other way around.) and heat the beaker **gently** on a hot plate. Bubbles of hydrogen gas ( $\text{H}_2(\text{g})$ ) should evolve. The cessation of  $\text{H}_2(\text{g})$  evolution signals the completion of the reaction; no more than 20–30 min of gentle heating should be needed. **Do not heat the reaction mixture vigorously and be sure that the beaker is covered with a watchglass throughout the heating period!** When the reaction is over, carefully remove the beaker from the hot plate and let the solution stand just until the beaker can be comfortably han-

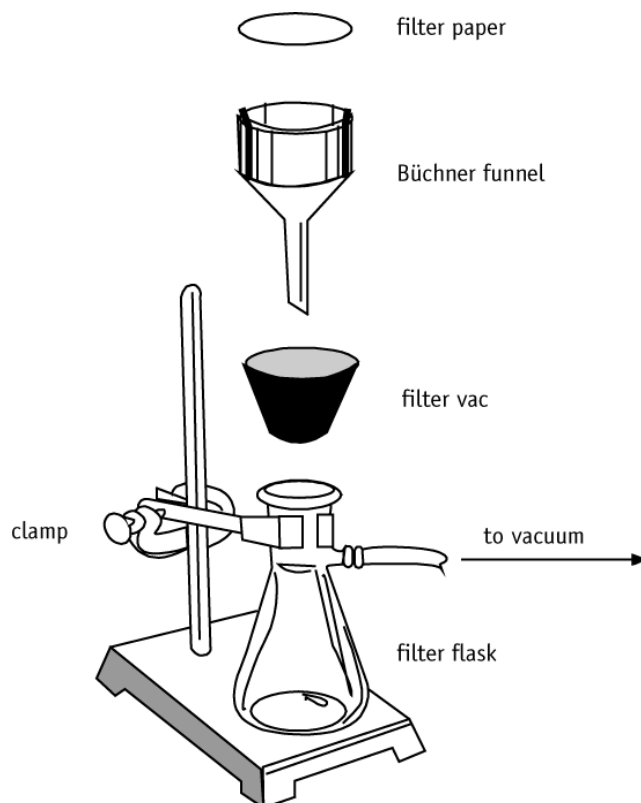
dled. While the reaction is in progress, set up the hot filtration described in the next section.

### Hot filtration

To remove any solid residue left over after the heating period, the reaction mixture must be filtered while it is still hot. Obtain a ring stand, a Büchner funnel, a filter flask equipped with a side-arm, heavy-walled tubing, a filtervac and filter paper. Make sure that the filter flask is clean; otherwise, wash it with detergent. The filter paper must lie perfectly flat on the floor of the funnel, yet cover all the holes.

Assemble the apparatus sketched in Figure 3-1, ensuring that the filter flask is securely clamped to a ring stand. Turn on the vacuum located on your bench top. Moisten the filter paper with a few drops of deionized water, swirl the beaker to mobilize the contents, and slowly pour a portion (not all of it) of the hot reaction mixture down a stirring rod into the funnel. Swirl

**Figure 3-1** Apparatus for hot filtration.



the beaker once again to mobilize the contents and pour another portion of the reaction mixture down a stirring rod into the funnel. Continue in this manner until all the reaction mixture has been filtered. Rinse the beaker with a 10-mL portion of deionized water and pour the rinse through the residue on the filter paper. Do not rinse the beaker with any more water than the amount specified: adding too much water at this point will make recovery of the final product difficult. Turn off the vacuum.

The liquid that collects in the filter flask (called the “filtrate”) should be perfectly clear and all solid residue should be trapped on the filter paper. Yes, if the filtrate is not completely free of suspended solid, the solution must be filtered again.

### **Formation of alum**

Transfer the filtrate from the filter flask to a clean 250-mL beaker. Rinse the filter flask with 10 mL of deionized water and add the rinse to the beaker. Again, do not rinse the beaker with any more water than the amount specified. In a small plastic tub, prepare an ice–water bath by combining equal portions of ice and tap water. Don’t make the ice–water bath too deep: you are going to set a beaker in it and you don’t the beaker to tip over.

Place the beaker holding the reaction mixture in the ice–water bath and allow the beaker to cool for about 5 min, then slowly, carefully and with constant stirring, add 20 mL of 9 *M* aqueous sulfuric acid ( $\text{H}_2\text{SO}_4(\text{aq})$ ) portion-wise to the cooled solution. Sulfuric acid causes severe burns: **wear heavy gloves during this operation**. You should notice the appearance of a milky white gel of aluminum hydroxide ( $\text{Al}(\text{OH})_3(\text{s})$ ). After all the  $\text{H}_2\text{SO}_4(\text{aq})$  is added, warm the solution gently on a hot plate until all the gel dissolves.

### **Isolation of the product**

Cooling the reaction mixture hastens the formation of alum crystals. Set the beaker containing the reaction mixture into an ice–water bath. Crystals of alum should begin to form. Allow the mixture to chill for about 10 min. If crystals do not form after 10 min, vigorously scratch the bottom of the beaker with

a metal spatula: this procedure provides a rough surface on which crystals can grow.

Clean and reassemble the suction filtration apparatus sketched in Figure 3-1; use a new piece of filter paper. Filter the alum crystals by suction from the chilled solution. In order to maximize recovery of alum, you may need to rinse the alum out of the beaker using no more than 10 mL of water. Allow the vacuum to pull air through the crystals for about 10 min or until the crystals are dry, fluffy and no longer clump together. While you are drying the alum in this way, carefully chop up the solid using a spatula: this operation will maximize the exposure of the crystals to air.

Use a spatula to transfer the air-dried alum crystals from the Büchner funnel to a watchglass, a small beaker, or a weighing boat that you have previously weighed to two decimal places. Record the mass of alum you synthesized to two decimal places. Present the alum to the TF. If the TF deems the alum satisfactory, place the alum in the alum storage bottle. Dispose of all solutions in a hazardous-waste receptacle.

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Lab report form

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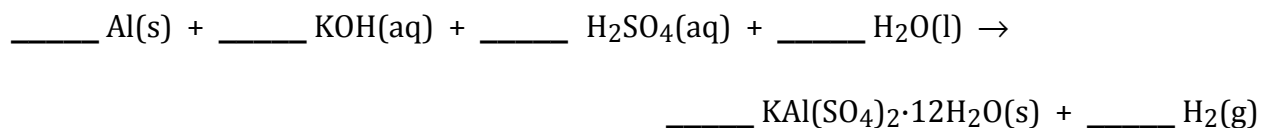
(I) Report the mass of aluminum taken and the mass of alum synthesized.

Mass of Al(s) taken = \_\_\_\_\_ g

Mass of alum obtained = \_\_\_\_\_ g

*Post-lab questions*

(1) Balance the equation of the synthesis of alum by filling in the appropriate coefficients.



(2.a) Calculate of the number of moles of Al(s) you used.

(2.b) Calculate of the number of moles of KOH you used.

(2.c) Calculate of the number of moles of H<sub>2</sub>SO<sub>4</sub> you used.

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(3) Given the number of moles of Al(s), KOH and H<sub>2</sub>SO<sub>4</sub> you calculated in (2), given that H<sub>2</sub>O is present in large excess, and given the balanced equation of the formation of alum, which substance is the limiting reagent? Show calculations and explain in words how you arrived at your answer. (Hint: See Appendix B “Stoichiometric Calculations” of this lab manual.)

(4) What is the percent yield of alum that you obtained? Show all calculations. (Hint: See Appendix B “Stoichiometric Calculations” of this lab manual.)



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### Lab report form

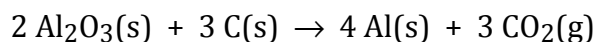
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(5) Most students report a percent yield of less than 100%. Which experimental operations contribute most to not achieving a 100% yield?

(6) Some students report a percent yield of greater than 100% most likely because of an experimental operation that was not done properly. Which incorrectly performed experimental operation would contribute most to obtaining a percent yield greater than 100%?

*The two questions that follow deal with the economics of aluminum recycling. Question (8) in particular shows how recycling can convert a 5¢ piece of trash (an empty soda can) into a higher-value commodity.*

(7) Aluminum is manufactured from aluminum oxide ( $\text{Al}_2\text{O}_3(\text{s})$ ) by the Hall-Héroult process:



Calculate the cost of producing a 17-g aluminum soda can given that (1) 18.36 kilojoule of electrical energy is required to produce 1 g of aluminum; (2) electricity costs 15 cents per kilowatt·hour; (3) 1 kilojoule = 1 kilowatt·sec.

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(8) Very pure alum is very expensive; however, the alum you synthesized is probably not very pure, so it would sell for only about 0.44 cents per gram. Calculate the dollar value of the alum obtained when a 17-g aluminum soda can is completely converted to alum.