ALUM FROM WASTE ALUMINUM CANS

INTRODUCTION

Modern beverage containers are usually composed of either aluminum, in the form of aluminum cans, or polyethylene terephthalate (PETE), the clear plastic beverage bottles. Approximately 300 million aluminum beverage cans are produced each day in the U.S. Aluminum is one of the most indestructible materials used in metal containers. The average "life" of an aluminum can is about one hundred years. Although aluminum is the third most abundant element in the earth's crust, the expense of extracting it from common soils is too expensive and the major source is the ore *bauxite*, the hydrated form of aluminum oxide, $Al_2O_3 \cdot 2H_2O$. Although there is concern regarding the depletion of aluminum ores, the major concern is the amount of electrical energy needed to extract the aluminum from its ores. To produce a single can, the energy needed is about the same as that required to keep a 100-watt bulb lit for 6 hours. That energy can be reduced by up to 95 percent by recycling used aluminum cans. Recycling also has the benefit of reducing litter from discarded cans and a number of states have passed laws requiring a deposit on aluminum cans to encourage recycling.

In this experiment, instead of recycling aluminum into new metal cans, a chemical process will be used that transforms scrap aluminum into a useful chemical compound, potassium aluminum sulfate dodecahydrate, $KAl(S0_4)_2 \bullet 12H_20$, commonly called "alum". Alum is widely used in the dyeing of fabrics, in the manufacture of pickles, in canning some foods, as a coagulant in water purification and waste-water treatment plants, and in the paper industry.

The class of chemical compounds known as "alums" are ionic compounds that crystallize from solutions containing sulfate anion, SO_4^{2-}, a trivalent cation, such as $A1^{3+}$, Cr^{3+}, or Fe^{3+}, and a monovalent cation, such as K^+, Na^+, or NH_4^+. Most alums crystallize readily as octahedra or cubes which, under the appropriate conditions, may grow to considerable size. Six of the 12 water molecules per formula unit are bound tightly to the trivalent cation. The remaining six are loosely bound to the sulfate anion and monovalent cation.

BACKGROUND INFORMATION

Although aluminum is a "reactive" metal, it reacts only slowly with dilute acids because its surface is normally protected by a very thin, impenetrable coating of aluminum oxide. (Such metals are referred to as self-protecting metals.) Alkaline solutions, or bases, (containing OH^-) dissolve the oxide layer and then attack the metal. Thus, in aqueous alkaline medium, aluminum is oxidized to the tetrahydroxoaluminate(III) anion which is stable only in basic solution.

$$2\ A1_{(s)} + 6\ H_2O_{(l)} + 2\ KOH_{(aq)} \rightarrow 2\ K[Al\ (OH)_4]_{(aq)} + 3\ H_{2(g)}$$

When sulfuric acid is slowly added to an alkaline solution of this complex anion, initially, one hydroxide ion is removed from each tetrahydroxoaluminate anion causing the precipitation of white, gelatinous aluminum hydroxide, $Al(OH)_3$,

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

The excess potassium hydroxide is neutralized by some of the sulfuric acid to form potassium sulfate.

$$2\ KOH_{(aq)} + H_2SO_{4(aq)} \rightarrow K_2SO_{4(aq)} + 2\ H_2O_{(l)}$$

On addition of more sulfuric acid, the aluminum hydroxide dissolves forming the hydrated aluminum cation

$$2\ A1(OH)_{3(s)} + 3\ H_2SO_{4(aq)} \rightarrow Al_2(SO_4)_{3(aq)} + 6\ H_2O_{(l)}$$

Addition of alkali to the $Al(OH)_3$ precipitate will also bring about dissolution by reforming $[Al(OH)_4]^-$. A hydroxide, such as aluminum hydroxide, that can be dissolved by either acid or base is said to be *amphoteric.*

When the acidified aluminum sulfate solution is cooled, potassium aluminum sulfate dodecahydrate ("Alum") precipitates.

$$A1_2(SO_4)_{3(aq)} + K_2SO_{4(aq)} + 24\ H_2O_{(l)} \rightarrow 2\ K[Al(SO_4)_2]\bullet 12H_2O_{(s)}$$

The overall reaction that takes place is the sum of the previous reactions.

$$2\ Al_{(s)} + 2\ KOH_{(aq)} + 4\ H_2SO_{4(aq)} + 22\ H_2O_{(l)} \rightarrow 2\ KA1(SO_4)_2\bullet 12H_2O_{(s)} + 3\ H_{2(g)}$$

SAFETY

Goggles or safety glasses must be worn at all times in the laboratory

Potassium hydroxide solutions are caustic. In the event of contact with your skin or eyes, wash the affected area immediately with lots of water. If necessary, seek qualified medical assistance.

Sulfuric acid is corrosive. In the event of contact with your skin or eyes, wash the affected area immediately with lots of water. If necessary, seek qualified medical assistance.

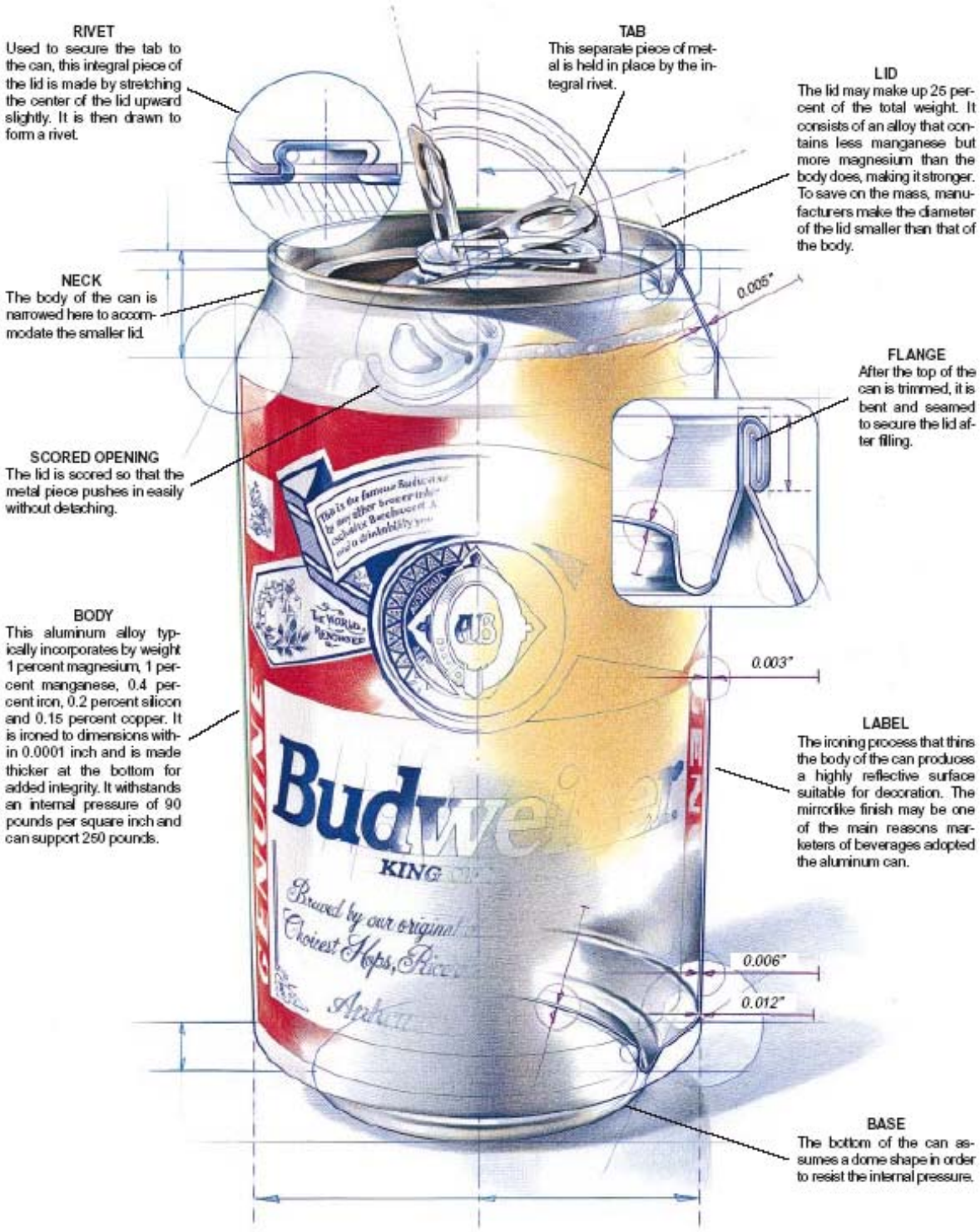
Ethanol is a flammable liquid. Avoid flames. Prolonged skin exposure can cause drying and cracking of the skin.

The aluminum metal may have shape edges. Exercise care in handling the metal.

Concentrated hydrochloric acid (Part 2 of this experiment) is corrosive and can cause severe chemical burns. Clean up any spills – if necessary, ask your instructor for assistance. In the event of skin contact, wash the affected area with water.

DISPOSAL

Dispose of all materials in the proper waste containers as provided in the laboratory.



ANATOMY OF MODERN BEVERAGE CAN reveals the dimensions that design and engineering must achieve on a daily basis. The goal of can makers is to reduce the amount of aluminum needed without sacrificing structural integrity. A can now weighs about 0.48 ounce; the industry hopes to reduce that weight by about 20 percent.

PART 1. PREPARATION OF POTASSIUM ALUMINUM SULFATE, $KAl(SO_4)_2 \cdot 12\ H_2O$ (ALUM)

MATERIALS NEEDED

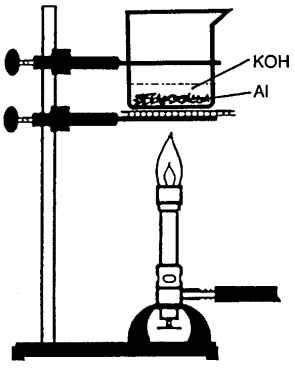
Aluminum beverage can
Potassium hydroxide, KOH, 1.4 M solution
Sulfuric acid, H_2SO_4, 9 M solution
Ethanol
Sandpaper
Scissors or metal snips
Ruler
Beakers, 50-mL or 100-mL, 250-mL, 600-mL
Bunsen burner or hotplate
Vacuum filtration apparatus: Buchner funnel, side-arm flask, rubber tubing, and filter paper
Stirring rod
Spatula
Graduated cylinder

PROCEDURE

If available, bring an empty aluminum beverage can to lab. If you cannot bring one, your instructor will provide one.

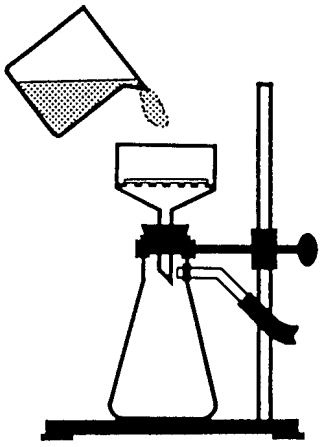
Using scissors or metal snips, cut a piece of aluminum approximately 5 cm x 7.5 cm from the can. Using a piece of sandpaper, scrape off any paint and/or plastic coating from both sides, as completely as possible. Weigh the cleaned piece of aluminum. You need approximately 1.0 g of aluminum (if the mass is between 0.9 and 1.2 g, that is acceptable).

Weigh a 250-mL beaker. Cut your aluminum sample into small squares of about 0.2 cm length (small pieces will react at a faster rate) and place them in the 250-mL beaker. Weigh the beaker and final sample to the nearest 0.01 g and record the mass. Determine the mass of the aluminum.



Add 50 mL of 1.4 M potassium hydroxide to the 250-mL beaker containing the aluminum pieces. Place the beaker on a hotplate, or ring stand and ring supports, in a fume hood and heat it so it is hot, but not boiling. Bubbles of hydrogen should form from the reaction between aluminum and aqueous potassium hydroxide. If the liquid level in the beaker drops to less than half of its original volume, add distilled water to maintain the volume at approximately 25 mL. The reaction is complete when the hydrogen evolution ceases and there are no visible pieces of aluminum metal. The final volume of the liquid should be about 25 mL If the reaction is not complete in about 30 minutes, check with your instructor.

During the reaction, the initially colorless mixture will turn dark gray or black. Pieces of plastic lining, that was not completely removed may be floating in the liquid. The dark material probably comes from the decomposition of residual paint or plastic lining. Also, note the periodic rise and fall of aluminum fragments during the reaction. Suggest an explanation.

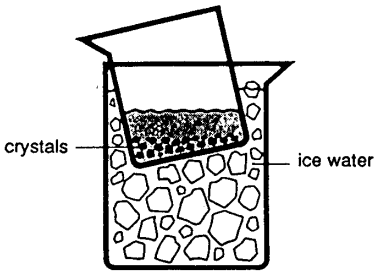


Set up a vacuum filtration apparatus. Be sure that the filter flask is securely clamped and the filter paper is moistened before you begin. Filter the hot solution to remove any solid residue. The filtrate should be clear with any dark residue left on the filter paper. Rinse the beaker twice with 5-mL portions of distilled water, pouring each rinse through the filter residue.

When all of the liquid has passed through the filter paper, break the vacuum by disconnecting the rubber tubing from the filter flask. Turn off the aspirator only after the vacuum has been broken.

Transfer the clear filtrate into a clean 250-mL beaker. Rinse the filter flask with 10 mL of distilled water and pour the rinse water into the beaker. If the filtrate is not yet cool, place the beaker in a cooling bath of cold water.

Slowly *and carefully, with stirring,* add 20 mL of 9.0 M H_2SO_4 to the cooled solution. The solution will get hot from the neutralization reaction occurring. You may notice the appearance of a white precipitate of aluminum hydroxide. Addition of the last few milliliters of the sulfuric acid will usually dissolve the $Al(OH)_3$. If necessary, warm the solution gently, while stirring, to completely dissolve any $A1(OH)_3$ that might have formed. The final solution will contain potassium ions (from the KOH used), aluminum ions, and sulfate ions. If, after a few minutes of heating, any solid residue remains, filter the mixture and work with the clear filtrate.



Prepare an ice-water bath by filling a 600-mL beaker two-thirds full with ice. Add cold water to just cover the ice. Set the reaction beaker into the ice-water bath to chill. Allow the mixture to chill thoroughly for about 15 minutes. Crystals of the alum should begin to form in a few minutes. If crystals do not form, you may have to induce crystallization. To induce crystallization, try stirring the solution rapidly, but do not splash any of the liquid from the beaker, or you may scratch the inside bottom of the beaker containing the solution with your stirring rod. As an alternative, you may add one or two very minute seed crystals. Seed crystals (if desired) can be obtained by placing a drop of solution on the end of a stirring rod and blowing on it until it is dry. As a last resort only, reduce the volume of solution by boiling away some of the water and then cooling the solution in the ice bath.

Clean and reassemble the vacuum filtration apparatus.

Mix 12 mL ethanol with 12 mL water in a small beaker. Remove the chilled solution of alum crystals from the ice bath and chill the ethanol mixture.

Filter the alum crystals from the chilled solution, transferring as much of the crystalline product as possible to the funnel.

Use half of the chilled ethanol solution to rinse the remaining crystals from the beaker into the funnel. Rinse the beaker again with the second half of the solution. Use a spatula to distribute the crystals evenly on the filter paper. Allow the aspirator to pull air through the crystals for about 10 minutes. (Ethanol in the wash solution reduces the solubility of the alum.)

While the crystals are drying, weigh a clean, dry 250-mL beaker to the nearest 0.01 g. Record this mass. Use your spatula to transfer all of the air-dried crystals from the filter paper into the beaker. Reweigh the beaker and the crystals. Record the mass. Determine the mass of the alum crystals.

Show the beaker containing your alum to your instructor, report the mass of alum obtained, and request a "product inspection." If your instructor considers your alum to be satisfactory, transfer the alum into a container supplied by your instructor. Save the alum to grow crystals of the product.

CALCULATIONS

Theoretical Yield

The theoretical yield, sometimes called the expected yield, is the amount of alum you would obtain from your starting mass of aluminum if all the reactions work perfectly and you are able to obtain all the intermediate compounds and products. The theoretical yield can be calculated from the overall reaction that takes place:

$$2\ Al_{(s)} + 2\ KOH_{(aq)} + 4\ H_2SO_{4(aq)} + 22\ H_2O_{(l)} \rightarrow 2\ KA1(SO_4)_2 \bullet 12H_2O_{(s)} + 3\ H_{2(g)}$$

According to the reaction, 2 moles of aluminum will react to form 2 moles of alum.

To calculate the theoretical yield of the alum, use the equation:

$$\textit{Theortical yield} = \text{Mass of aluminum used} \times \frac{\text{1 mole aluminum}}{\text{atomic weight aluminum}} \times \frac{\text{moles of alum produced}}{\text{moles of aluminum used}} \times \frac{\text{formula weight of alum}}{\text{1 mole alum}}$$

where the middle term of the equation is the mole ratio of *moles of alum produced* to *moles of aluminum used* from the balanced equation, above.

Percent Yield

The percent yield is the percent of the theoretical yield you actually obtained. To calculate the percent yield, use the equation:

$$\text{Percent yield} = \frac{\text{Mass of alum obtained}}{\text{Theoretical yield of alum}} \times 100\%$$

PART 2. VERIFICATION OF POTASSIUM ALUMINUM SULFATE

MATERIALS NEEDED

Alum, prepared in Part 1 of this experiment
0.5 M $BaCl_2$ solution in a dropper bottle
0.15 M KOH solution in a dropper bottle
Hydrochloric acid, concentrated
Spatula or scoopula
Test tubes, 12 x 75 mm
Beaker, 50 mL
Watch glass
pH paper
Nichrome wire test loop
Bunsen burner

PROCEDURE

A. Test for Sulfate Ion

Use your spatula to transfer a small amount of your alum, prepared in Part 1, to a small test tube. Add approximately 1 mL of distilled or deionized water. Agitate the test tube gently to dissolve the alum crystals.

Test the solution with a piece of pH paper. The solution should be acidic (pH less than 7).

Add 1 drop of 0.5 M $BaCl_2$ to the solution in the test tube. If sulfate ion is present, a white precipitate will be formed.

B. Test for Potassium

Obtain a nichrome wire test loop.

Heat the nichrome wire loop in a Bunsen burner flame. If a colored flame is produced, you will need to clean the wire test loop.

To clean the wire test loop, do this procedure under a fume hood.

Pour about 10 mL of concentrated hydrochloric acid into a clean, dry 50-mL beaker.

Heat the test loop in a Bunsen burner flame until it is red hot. Quickly, place the hot test loop into the concentrated hydrochloric acid. This will dissolve most of the chemicals that are adhering to the test loop. (It may be necessary to repeat this several times to completely clean the test loop. Do not discard the hydrochloric acid until the test loop is clean.)

Heat the test loop in a Bunsen burner flame. If a colored flame is produced, clean the test loop in the concentrated hydrochloric acid. If necessary, repeat this procedure until the test loop is clean and does not produce a colored flame.

Place a small amount of your alum crystals on a watch glass. Heat the clean nichrome test loop in a Bunsen burner flame until it is red hot, then quickly touch the hot loop to the alum crystals on the watch glass. Some crystals should stick to the test loop.

Slowly, bring the test loop with the crystals toward the burner flame. What do you observe as the crystals come in contact with the flame?

Heat the test loop and crystals in the burner flame for about 5 seconds until they glow red. Remove the loop from the flame and allow it to cool.

Place 1 mL of distilled or deionized water to a small test tube. Insert the test loop with the cooled crystals into the test tube and stir gently to dissolve the residue.

Remove the test loop and add 1 drop of the 0.5M $BaCl_2$ solution. What do you observe? Why are your observations different from the results in Part A, above?

C. Test for aluminum

Use your spatula to transfer a small amount of your alum, prepared in Part 1, to a small test tube. Add approximately 1 mL of distilled or deionized water. Agitate the test tube gently to dissolve the alum crystals.

Add 1 drop of 0.15 M KOH to the solution in the test tube. If aluminum is present, a white gelatinous precipitate will be formed.

REPORT FORM

ALUM FROM WASTE ALUMINUM CANS

Name _____ Course/Section _____

Partner's Name (if applicable) _____ Date _____

PART 1. PREPARATION OF POTASSIUM ALUMINUM SULFATE, $KAl(SO_4)_2 \cdot 12\ H_2O$ (ALUM)

DATA AND RESULTS

Mass of 250-mL beaker _____ g

Mass of 250-mL beaker and aluminum _____ g

Mass of aluminum used _____ g

Mass of clean, dry 250-mL beaker _____ g

Mass of 250-mL beaker and alum _____ g

Mass of alum obtained _____ g

Calculate the theoretical yield of the alum based on the mass of aluminum you used:

Calculate the percent yield of the alum:

PART 2. VERIFICATION OF POTASSIUM ALUMINUM SULFATE

DATA AND RESULTS

A. Test for Sulfate Ion

Is the solution of alum acidic? What is the approximate pH of the solution?

Was there a reaction with 0.5 M $BaCl_2$ solution? Write a balanced chemical reaction for the change that occurred.

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B. Test for Potassium

What did you observe when the alum crystals came in contact with the flame? What does your result indicate?

After heating the alum crystals in the flame and testing the resulting solution with 0.5M $BaCl_2$ solution, what did you observe? Why are your observations different from the results in Part A?

C. Test for Aluminum

Was there a reaction with 0.15 M KOH solution? Write a balanced chemical reaction for the change that occurred.

QUESTIONS

1. Why is the inside of an aluminum can lined with a plastic coating?

2. Why is the percent yield of alum usually less than 100%? (What happened to the missing material?)

3. Is this process an effective method for the recycling of aluminum?

4. Do the verification tests in Part 2 of the experiment prove that you made alum? Explain.