THE SYNTHESIS OF
POTASSIUM ALUMINUM SULFATE (Alum)
FROM ALUMINUM SCRAP

This experiment has the following objectives:
1. To be aware of the need for recycling solid wastes, particularly scrap metal like aluminum.
2. To learn some of the chemistry of aluminum.
3. To become familiar with the use of laboratory equipment such as beakers, flasks, Bunsen burners, and so on.
4. To be able to perform the techniques of weighing, gravity and vacuum filtration and crystallization.
5. To be able to apply a knowledge of the stoichiometry of a sequence of chemical reactions to the calculation of the percentage yield of alum synthesized from aluminum scrap.

INTRODUCTION

Modern societies have concentrated on what might be considered the most primitive of methods of solid waste disposal — burning or burying! This type of behavior is a strange anomaly, because solid waste is probably the oldest of man’s pollutants and yet has only recently received serious attention from scientists and technologists.

Like all other environmental problems, solid waste problems are intensified by modern civilization—more people and more things. In 1920 the average American generated 2 3/4 pounds of solid waste per day; in 1970 the average was 5 pounds per day and by 1980 it was about 8 pounds per day! The composition of all this solid waste is shown in the TABLE on the next page; it is drawn from a Public Health Service study of municipal refuse for 1966-1968.

Unfortunately, the problem is compounded by the fact that one of the most important types of resource at the base of our technological society is metal. Metals such as copper, chromium, molybdenum, tin, zinc, tungsten, and aluminum have become as necessary to economics as water and fossil fuels. Aluminum is a classic example!

Aluminum is the third most abundant element, and most abundant metal, in the Earth’s crust. It is concentrated in a number of high-grade, natural bauxite deposits, almost all of which are located outside of the U.S. Changes in the international situation (the so-called Third World owns most of the
The depletion of high grade ores, bauxite), and the depletion of high grade ores, is forcing the aluminum industry to use lower grade ores with correspondingly higher prices for aluminum and products made from it. At the same time this is happening, the use of aluminum in disposable products (e.g., beverage cans, foil, etc.) is increasing enormously. Even the most solid of all solid wastes, the automobile, now contains more aluminum alloys. The stimuli to recycle aluminum are thus very strong and are being reinforced by other recent developments. The production of aluminum from natural sources like bauxite (aluminum oxide, Al2O3) and cryolite (Na3AlF6) involves an electrolytic process which uses large amounts of electricity. The sky-rocketing cost of that form of energy is well known! The energy costs of recycling aluminum metal, by shredding, melting and casting, are a small fraction (about 5-10%) of the energy cost of producing the metal from ore.

Finally, in an ironic twist, one of the properties of aluminum that is responsible for its current, wide-spread use can cause a serious environmental problem. Aluminum does not corrode as does iron and steel. A fresh aluminum surface reacts very rapidly with oxygen to produce an oxide coating that is so

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**SOLID WASTE PROFILE—1965 to 1968**

<table>
<thead>
<tr>
<th>Item</th>
<th>% by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paper products</td>
<td>55.0</td>
</tr>
<tr>
<td>Glass</td>
<td>7.7</td>
</tr>
<tr>
<td>Metals</td>
<td>6.8</td>
</tr>
<tr>
<td>Vegetable scraps</td>
<td>2.9</td>
</tr>
<tr>
<td>Meat scraps</td>
<td>2.9</td>
</tr>
<tr>
<td>Other foods</td>
<td>2.9</td>
</tr>
<tr>
<td>Leaves, grass, etc.</td>
<td>10.0</td>
</tr>
<tr>
<td>Other</td>
<td>2.8</td>
</tr>
<tr>
<td>Adjusted moisture</td>
<td>9.0</td>
</tr>
</tbody>
</table>

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**Figure** An electrochemical cell used to produce aluminum metal. The aluminum, in the form of compounds such as Al2O3 in Na3AlF6 is electrolyzed (reduced) at a graphite cathode. Because the cell is at a high temperature, the aluminum is molten, and is drawn off the bottom of the cell. See Chemistry & Chemical Reactivity, 4th edition, page 992.

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tenacious and impervious that no further reaction takes place. The thin layer is not necessary. Hence, skyscraper facings, airplanes, Airstream trailers, and beer cans can be nice shiny objects. The discarded aluminum can has become nearly immortal! It has an average “lifetime” in the environment of greater than 100 years.

There are now a number of successful recycling programs for aluminum cans. Collection centers are paying around 15 cents per pound for scrap aluminum. The problem of recycling cans that end up in the municipal landfill has not yet been solved, but progress is being made in designing large scale separators to separate aluminum, steel, glass, and paper from trash. The scrap aluminum is usually shredded, melted down, cast, and eventually made into an aluminum product.

In this experiment you will be recycling aluminum scrap in a very unusual way, and you will produce two products which are potentially very useful: hydrogen gas ($H_2$) and very pure potassium aluminum sulfate ($KAl(SO_4)_2\cdot12H_2O$ or alum). Hydrogen gas has great potential use as a fuel, if some of its dangerous properties can be controlled (mixtures of $H_2$ and air are highly explosive). Hydrogen gas, when burned properly, produces a large amount of heat and no pollution, the only combustion product being water. Alum is a widely used chemical in industry, playing an important role in the production of many products used in the home and industry. The pulp and paper industry alone consumes 70% of the more than one million tons of alum produced annually in the U.S. It is used to “size” the paper. The second largest use is in the purification of water for human and industrial consumption. Other uses include soaps, greases, fire extinguisher compounds, textiles, leather, synthetic rubber, drugs, cosmetics, cement, plastics, and pickles.

It is important to note here that this particular process for converting aluminum into alum would produce very expensive alum! Alum can be made, very cheaply at the moment, using clay as the raw material. Consequently, the procedure used in this experiment is not used as an industrial method for recycling aluminum.

**THE CHEMISTRY OF THE EXPERIMENT**

Aluminum beverage cans generally have a thin coating of plastic on the inside that protects the aluminum from the corrosive action of the chemicals in the beverage. The outside usually has a thin coating of paint. These coatings must be removed before any chemical reactions with the metal can be carried out. The coatings may be effectively scraped off with a metal pan cleaner. A cleaned piece of metal is then dissolved in a potassium hydroxide solution according to the following complete, balanced equation:

$$2\text{Al(s)} + 2\text{KOH(aq)} + 6\text{H}_2\text{O(liq)} \rightarrow 2\text{KAl(OH)}_4\text{(aq)} + 3\text{H}_2\text{(g)}$$

or the net ionic equation

$$2\text{Al(s)} + 2\text{OH}^-\text{(aq)} + 6\text{H}_2\text{O(liq)} \rightarrow 2\text{Al(OH)}_4^-\text{(aq)} + 3\text{H}_2\text{(g)}$$

The dissolution of Al(s) in aqueous KOH is an example of an oxidation-reduction or redox reaction. [The Al metal is oxidized to aluminum with an oxidation number of +3 and the hydrogen in KOH or in water is reduced from an oxidation number of +1 to zero in hydrogen gas.] The $\text{Al(OH)}_4^-$ ion is a com-
plex ion called “aluminate.” These reactions illustrate the reason that alkaline products (such as detergents, cleaners, shampoos, and so on) are never stored in an aluminum container. The aluminum would slowly dissolve!

After filtration to remove residual plastic and paint decomposition products, the alkaline solution of Al(OH)\(_4^-\) is clear and colorless. The H\(_2\) is evolved as a gas and mixes with the atmosphere. The chemical species in solution are potassium ions (K\(^+\)) and aluminate ions [Al(OH)\(_4^-\)] ions (plus any unreacted KOH).

Sulfuric acid is now added and two sequential reactions occur. Initially, before the addition of all the acid, the complete reaction is

\[ 2 \text{KAl(OH)}_4\text{(aq)} + \text{H}_2\text{SO}_4\text{(aq)} \rightarrow 2 \text{Al(OH)}_3\text{(s)} + 2 \text{H}_2\text{O}\text{(liq)} + \text{K}_2\text{SO}_4\text{(aq)} \]

or the net ionic equation

\[ \text{Al(OH)}_4^-\text{(aq)} + \text{H}^+\text{(aq)} \rightarrow \text{Al(OH)}_3\text{(s)} + \text{H}_2\text{O}\text{(liq)} \]

The reaction above is an acid-base reaction in which the \text{H}^+\text{ ions from the sulfuric acid neutralize the base Al(OH)\(_4^-\)} to give a thick, white, gelatinous precipitate of aluminum hydroxide, Al(OH)\(_3\).

As more sulfuric acid is added, the precipitate of Al(OH)\(_3\) dissolves.

\[ 2 \text{Al(OH)}_3\text{(s)} + 3 \text{H}_2\text{SO}_4\text{(aq)} \rightarrow \text{Al}_2\text{(SO}_4\text{)}_3\text{(aq)} + 6 \text{H}_2\text{O}\text{(liq)} \]

or the net ionic equation is

\[ \text{Al(OH)}_3\text{(s)} + 3 \text{H}^+\text{(aq)} \rightarrow \text{Al}^{3+}\text{(aq)} + 3 \text{H}_2\text{O}\text{(liq)} \]

to give aluminum ions, Al\(^{3+}\), in solution. The solution at this point contains Al\(^{3+}\) ions, K\(^+\) ions (from potassium hydroxide), and SO\(_4^{2-}\) ions (from sulfuric acid). On cooling, crystals of hydrated potassium aluminum sulfate, KAl(SO\(_4\))\(_2\)•12 H\(_2\)O (or alum) are very slowly deposited. In the experiment the crystallization process is speeded up by providing a small “seed crystal” of alum for the newly forming crystals to grow on. Cooling is needed because alum crystals are soluble in water at room temperature. The complete equation is

\[ \text{Al}_2\text{(SO}_4\text{)}_3\text{(aq)} + \text{K}_2\text{SO}_4\text{(aq)} + 24 \text{H}_2\text{O}\text{(liq)} \rightarrow 2 \text{KAi(SO}_4\text{)}_2\text{•12 H}_2\text{O} \]

and the net ionic equation is

\[ \text{K}^+\text{(aq)} + \text{Al}^{3+}\text{(aq)} + 2 \text{SO}_4^{2-}\text{(aq)} + 12 \text{H}_2\text{O}\text{(liq)} \rightarrow \text{KAi(SO}_4\text{)}_2\text{•12 H}_2\text{O(s)} \]

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 but does not dissolve them. It also helps to dry the crystals quickly, because alcohol is more volatile than water.

**EXPERIMENTAL PROCEDURE**

1. Clean all glassware.
2. Pierce the can at the lower end of the side with the point of a pair of scissors. Cut around so that the sides of the can are cut out. Deposit the waste aluminum scraps left over in the box provided.
3. Lay the rectangular piece of aluminum (the sides of the can) on the bench and scour both sides with the pan scrubber provided. Make sure that an area of 2” x 2(1/4)" is clean on both sides.
4. Wipe the metal clean with a paper towel and cut out a clean piece that is about 2” x 2 1/4".
5. Take this piece to the analytical balance and weigh it. If the piece weighs more than 1 g, cut small pieces off until it weighs about 1 gram. Now weigh the piece accurately, and record the mass on your report form.

6. Cut the weighed piece into smaller pieces, and place them in a clean 250 mL beaker. Do not lose any metal bits.

7. Using a graduated cylinder, add 50 mL of 1.4 M KOH. DO IT IN THE HOOD!

8. Set up a ring stand, ring clamp, gauze, and bunzen burner. Place the beaker on the gauze and heat the beaker on a low flame. It is not necessary to boil it. The aluminum will take about 20 minutes to dissolve.

9. While it is dissolving, set up an apparatus for gravity filtration (as described by the figure on page A-3). Place a clean funnel, with a piece of folded filter paper, on a 125 mL Erlenmeyer flask.

10. When the aluminum has dissolved (as evidenced by the lack of bubbles of H₂ gas given off), gravity filter the solution. Only fill the funnel to within 1/2" 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.

11. Allow the flask to cool. While it is cooling, wash the funnel and beaker with lots of tap water to remove any potassium hydroxide.

12. When the solution is reasonably cool, add 20 mL of 9 M H₂SO₄ (with a graduated cylinder) quickly and with care. It is important that you swirl the flask as you add the acid. The solution will get quite warm. If there are any white flecks left in the solution after the addition of the H₂SO₄, place the flask on the Bunsen burner apparatus and warm it with swirling until all of the solid material has dissolved.

13. Make an ice bath by putting ice and water into a 600 mL beaker.

14. Allow the flask to cool a little and then place it in the ice bath and allow it to cool for an additional 5 minutes.

15. 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.

16. While the solution is cooling, pour 50 mL of 50% alcohol/water mixture into a test tube and place it in the ice bath to cool.

17. Set up the vacuum filtration apparatus (Buchner funnel) as illustrated by your instructor and on the following page. Pour some distilled water onto the filter.

18. 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. The water aspirator should be kept going all through this process.

19. 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.

For the procedure on gravity filtration, see the Crystal Growing experiment, page A-3. Use great caution in handling 9 M sulfuric acid. It is very corrosive.
20. Wash the alum crystals, now on the filter, with the cooled alcohol/water mixture using the following procedure: Disconnect the aspirator hose from the aspirator unit on the faucet. Pour about 5-10 mL of alcohol/water onto the crystals and gently stir. Reconnect the aspirator hose, and suck the crystals dry. After pouring on the last portion of alcohol/water suck the crystals dry for at least 20 minutes.

21. Place the nearly dry alum crystals in the evaporating dish in your desk until the next laboratory period.

22. In the next period accurately weigh your yield of crystals, recording the weight of product on the report form.

23. After weighing your alum sample (make sure you have recorded the mass), place the sample in your large test tube and hand it in to your instructor. Make sure that your test tube is labeled with your name and lab section number.
CALCULATIONS

The objectives of this experiment are:

a) to prepare a sample of alum from aluminum scrap, and
b) to perform some stoichiometry calculations, specifically the percent yield of product.

The stoichiometry involved in the sequence of reactions leading to the preparation of alum provides the mole relationship between aluminum and alum that is required to calculate the percentage yield.

\[
\begin{align*}
2 \text{Al(s)} + 2 \text{KOH} + 6 \text{H}_2\text{O} & \rightarrow 2 \text{K[Al(OH)]}_4 + 3 \text{H}_2 \quad (1) \\
2 \text{K[Al(OH)]}_4 + \text{H}_2\text{SO}_4 & \rightarrow 2 \text{Al(OH)}_3 + 2 \text{H}_2\text{O} + \text{K}_2\text{SO}_4 \quad (2) \\
2 \text{Al(OH)}_3 + 3 \text{H}_2\text{SO}_4 & \rightarrow \text{Al}_2(\text{SO}_4)_3 + 6 \text{H}_2\text{O} \quad (3) \\
\text{Al}_2(\text{SO}_4)_3 + \text{K}_2\text{SO}_4 + 24 \text{H}_2\text{O} & \rightarrow 2 \text{KAl(SO}_4)_2 \bullet 12\text{H}_2\text{O} \quad (4) \\
2 \text{Al(s)} + 2 \text{KOH} + 4 \text{H}_2\text{SO}_4 + 22 \text{H}_2\text{O} & \rightarrow 2 \text{KAl(SO}_4)_2 \bullet 12\text{H}_2\text{O} + 3 \text{H}_2 \quad (5)
\end{align*}
\]

The overall reaction for the synthesis of alum, equation (5), is obtained by adding reactions (1-4) and canceling like-species. The overall reaction stoichiometry (5) informs us that 2 moles of aluminum will produce 2 moles of alum.

Your calculations should proceed as follows:

a) Calculate the number of moles of Al used from the mass of Al used.

b) Knowing that the stoichiometric factor is

\[
\text{Stoichiometric factor} = \frac{2 \text{ mol KAl(SO}_4)_2 \bullet 12\text{H}_2\text{O}}{2 \text{ mol Al}}
\]

calculate the quantity of alum (KAl(SO}_4)_2 \bullet 12\text{H}_2\text{O}, in moles, that should be produced theoretically from the quantity (in moles) of aluminum metal used. Enter this calculation on your report form.

c) Knowing the number of moles of KAl(SO}_4)_2 \bullet 12\text{H}_2\text{O} expected, calculate the mass of KAl(SO}_4)_2 \bullet 12\text{H}_2\text{O} expected (the theoretical yield). Enter this on your report form.

d) Calculate the percentage yield of alum, if

\[
\% \text{ Yield} = \frac{\text{mass of alum obtained (g)}}{\text{mass of alum theoretically obtainable (g)}} \times 100\%
\]

Enter the result of your percent yield calculation on your report.

Give the completed report form to your laboratory instructor by the indicated due date. Be sure that your instructor also has the sample of the alum that you prepared.
SYNTHESIS OF POTASSIUM ALUMINUM SULFATE

Name ________________________________________ Section ________ Grade ______

EXPERIMENTAL DATA

Mass of aluminum used ________________________________

Mass of paper or beaker + KAl(SO₄)₂•12H₂O _________________

Mass of paper or beaker ________________________________

Mass of KAl(SO₄)₂•12H₂O ________________________________

CALCULATIONS: Show your calculations throughout!
1. How many moles of aluminum metal did you use?

2. Assuming that Al metal is the limiting reagent, what quantity of potassium aluminum sulfate (alum), in moles, is theoretically obtainable?

3. What mass of potassium aluminum sulfate (alum, KAl(SO₄)₂•12H₂O), in grams, is theoretically obtainable from your mass of Al? That is, what is the theoretical yield of alum?

4. What was the percentage yield of your product, KAl(SO₄)₂•12H₂O?