Chemistry of Aluminum

In the middle of the nineteenth century, aluminum was more expensive than silver or gold despite the fact that it is the third most abundant element on earth. In fact, people owned aluminum-ware as opposed to silverware to flaunt their wealth.

In 1885 a chemistry professor suggested to his freshman class that whoever extracted aluminum cheaply from the ore bauxite would be wealthy. Twenty year old student Charles Hall worked in a shed near his home for the next year until he developed a process of extraction of aluminum from bauxite.

The ore bauxite consists mainly of aluminum and iron oxides. In the Hall process, $\text{Al}_2\text{O}_3$ separated from $\text{Fe}_2\text{O}_3$ by dissolving the bauxite in aqueous NaOH. The insoluble $\text{Fe}_2\text{O}_3$ is removed, and the $\text{Al}_2\text{O}_3$ is recovered and dried. Pure aluminum can be prepared by electrolysis of molten $\text{Al}_2\text{O}_3$; but the process requires a tremendous amount of energy, first to melt the $\text{Al}_2\text{O}_3$ (mp 2045° C) and then for the endothermic electrolysis reaction. Hall found a solvent, cryolite $\text{Na}_3\text{AlF}_6$, with a lower melting point, (1000° C) that ionizes $\text{Al}_2\text{O}_3$ but is not itself oxidized or reduced during electrolysis. The electrolysis reactions are:

$$\text{Al}^{3+} + 3e^- \rightarrow \text{Al} \quad \text{(cathode)}$$

$$\text{C} + 20^2- \rightarrow \text{CO}_2 + 4e^- \quad \text{(anode)}$$

Today aluminum is certainly cheaper than silver or gold. However, the total energy needed to extract aluminum from bauxite is over 25 times greater than the energy needed to obtain aluminum by recycling. The recycling process keeps Al in the zero oxidation state. If Al were allowed to oxidize to $\text{Al}^{3+}$ during recycling, large amounts of energy would be required to reduce it again.

In this experiment you will convert aluminum foil into alum, $\text{KAl(SO}_4\text{)}_2(\text{H}_2\text{O})_{12}$. Alum has many uses-as a mordant in dyeing fabrics, in the manufacture of paper and cement, in baking powder, in preparing pickles, as a catalyst in the synthesis of NH$_3$. See The Merck Index for other uses.
1. Weigh about 0.50 g of sample to the nearest 0.01 gram. Add or subtract pieces of foil, as needed. Tear the foil into tiny pieces, less than one square centimeter.

2. Pure aluminum readily dissolves in dilute acid, but aluminum surfaces are usually covered by a thin layer of Al₂O₃. Both Al and Al₂O₃ are soluble in dilute base, forming Al(OH)₄⁻ ions. H₂ gas is also produced by Al. Put your weighed aluminum in a 150 mL beaker. (Take the beaker to HOOD, if there is room.) Be sure your goggles are on. Add 25 mL of 1.4 M KOH. A bubbling reaction should begin almost immediately. If this does not happen, warm the beaker gently on a hot plate until the shiny aluminum has dissolved (about 10 minutes or less). Caution: H₂ is explosive.

3. Gravity-filter the hot gray solution with a plastic funnel and fluted filter paper. Collect the filtrate in a 100 or 150 mL beaker. Rinse the beaker twice with about 5 mL portions of water from your water bottle, filter, and collect these rinsing, too. Discard the filter paper.

4. Goggles are still on! Slowly with stirring acidify the cooled solution with 10 mL of 9 M H₂SO₄. Test with blue litmus paper. (Blue litmus should turn pink.) Add a little more acid, if necessary, for the proper litmus test.

   Reactions: \[ \text{Al(OH)}_4^- + H^+ \rightarrow \text{Al(OH)}_3 \text{acid} \quad \text{white ppt} \]

   \[ \text{Al(OH)}_3 + H^+ \rightarrow \text{Al}^{3+} \text{more acid} \]

   CAUTION: Sulfuric acid burns the skin.

5. Evaporate the solution down to a volume of about 25 mL, if necessary, with the burner. Cool under tap water. (Store this solution in your locker until the next lab, if you wish to try to grow some crystals. When you return, if crystals are present, proceed to Step 8.)

6. Otherwise, chill the beaker in an ice-water bath for 15 to 20 minutes, or until you think crystals have stopped forming. Alum should crystallize. (What was the source of the K⁺, Al³⁺, and SO₄²⁻?) Set up your plastic funnel for filtering.

7. Decant (pour off the liquid) any liquid remaining in the beaker, and add about 10 mL of 50% ethanol to the solid residue. Mix them well, pour the slush into the plastic funnel
and gravity-filter. Rinse the beaker twice with 5 mL of chilled 50% ethanol, and add them through the filter. Finally, spread the product onto one of your large pieces of paper towel or filter paper, then press them with another piece to complete the drying. (An overnight air-drying may even be necessary.) Use the following reaction

\[
K(aq)\textsuperscript{+} + Al(aq)\textsuperscript{3+} + 2 SO\textsubscript{4}\textsuperscript{2–} + 12 H\textsubscript{2}O(l) \rightarrow KAl(SO\textsubscript{4})\textsubscript{2} \cdot 12H\textsubscript{2}O(l)
\]

to calculate your theoretical yield of alum.

8. Weigh the dry crystals. Calculate your per cent yield, based on the Al used.

9. Qualitative Chemical Tests

Run tests for the following ions on small amounts of your alum crystals (wet or dry).

   a. **Potassium.** Do a flame test for potassium as directed in the experiment “Expt 9 Flame tests.” It is not necessary to use any filters, though.

   b. **Aluminum.** Dissolve a few crystals of alum in approximately 1 mL water. Add a few drops of 6 M NH\textsubscript{3}. The formation of a white gel, Al(OH)\textsubscript{3}, indicates the presence of aluminum.

   c. **Sulfate.** Dissolve a few crystals of alum in approximately one mL water. Add a few drops of 0.1 M Ba(NO\textsubscript{3})\textsubscript{2}. The formation of a white precipitate of BaSO\textsubscript{4} indicates the presence of sulfate.

10. Save your crystals or put them in the jar for the next project.

**Growing Crystals**

Alum is a good choice for a first attempt at growing crystals, since alum crystals grow well. Each lab will grow a crystal for the next two weeks.

We will use an evaporation method for growing the crystal. A saturated solution is allowed to evaporate slowly, and as the water evaporates, the crystal continues to grow larger.

Transfer about 25 grams of alum into a 250 ml beaker. Add 175 ml of water. The solubility of alum is 1 gram per 7.2 ml of water. Warm and stir until the alum dissolves. If there is any residue, filter it out.

Tie a thread to a glass rod that is to lay across the beaker. Allow the thread to dip about 1 cm beneath the surface of the alum solution. Rub a little Vaseline on the thread above the solution to prevent crystals from growing in it. Cover the beaker with a paper towel.
Next lab pick out the best crystal, hopefully at least 2 mm long, to be used as a seed crystal. Tie the seed crystal with a slip knot, using a piece of thread. Suspend the thread from a wire bent as shown. Be sure the wire hook is well below the liquid surface. Cover the beaker with a paper towel held in place with a rubber band. Allow the crystal to grow undisturbed as long as possible.
Experiment 18

Name __________________
Lab __________________

g. Aluminum used ___________________
theoretical yield of Alum ______________________
Actual yield of Alum ______________________
% yield ______________________________

Qualitative test results for say whether it is positive or negative and write observations

<table>
<thead>
<tr>
<th>ion</th>
<th>Positive or negative(present or not present)</th>
<th>Observations that lead to your conclusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>K</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SO₄²⁻</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Question:

In a laboratory you have ZnCO₃, HCl, Cl₂, HNO₃, Zn(OH)₂, and Zn metal (also water), **you may not use a reaction more than once.**

Describe how to prepare zinc chloride and write the balance equation using the following types of reactions

a. an acid base reaction

b. a gas forming reaction

c. an oxidation-reduction reaction