ALUMINUM ADVENTURE
INTRODUCTION

Description
Students observe an electrochemical cell constructed with pieces of aluminum that serve as both the anode and cathode. Students then determine what effect an organic dye (Rit® brand dye) has on aluminum that has been anodized for 5, 10, or 15 minutes. They also investigate the effects of heating an anodized aluminum strip before treatment with the dye bath. Finally, students use their accumulated knowledge of the anodizing process to design and treat an art object.

Goals for This Experiment
The goals for this experiment are to have students:
1. anodize and dye a piece of aluminum;
2. compare three types of treated aluminum and their reactions to dye and to dilute HCl; and
3. use their accumulated knowledge of anodization to design and treat an art object.

Recommended Placement in the Curriculum
The Aluminum Adventure lab would be best implemented late in the second semester of General Chemistry Laboratory. Students do not need prior experience in electrochemistry, but the concepts of electrochemistry, oxidation, and reduction should be covered before the students hand in their lab reports. This ensures that students are able to think intelligently about what they did in lab.

ALUMINUM ADVENTURE

This laboratory investigation has three parts. You must complete the first part before you are given your task for the remaining parts. Once you complete Part I (described below), show your instructor the data you have collected and your responses to the questions asked. When your instructor agrees that you are ready, you will be provided with the next step, and so on.

PART I: What happens to the aluminum strip?

TASK

In this part of the investigation, you will use an electrochemical cell constructed with pieces of aluminum that serve as both anode and cathode. Your task is to observe the cell and its components before, during, and after operation to determine and quantify any changes that might occur to the aluminum anode as a result of the treatment. You will also prepare a brief statement hypothesizing what has occurred. Balances, magnifying lenses, and conductivity testers will be provided to aid you in your task. You will need to provide your instructor with the hypothesis, the experimental data to support it, and answers to the questions asked in the procedure.

GENERAL PROCEDURE

1. The electrochemical cell you will use is composed of the following parts.
   - ✔ The cathode is an aluminum sheet about 10 cm x 20 cm that has been rolled to fit inside a 400-mL beaker. The cathode (sheet) will be connected to the negative (-) side of the power supply. DO NOT MAKE THIS CONNECTION AT THIS TIME.

   - ✔ The electrolyte is 3.0 M sulfuric acid (3.0 M H_2SO_4).
     CAUTION: 3.0 M sulfuric acid is a strong acid. If it spills on you, rinse immediately with water. If it spills on the bench, wipe up the spill with water immediately.

   - ✔ The anode is an aluminum strip cut to 1 cm x 8 cm. It will be connected to the positive(+) side of the power supply. DO NOT MAKE THIS CONNECTION AT THIS TIME.
     CAUTION: The aluminum strips have sharp edges. Handle them with care.

2. It is important to clean the aluminum surface of any surface oil or dirt before it is used. The large aluminum sheet has been cleaned for you. You should leave this in the beaker during all of the procedures. You will, however, need to clean the aluminum piece that will be the anode. Do this by rinsing with acetone and rubbing dry with a paper towel. After cleaning the aluminum surface of surface oil and dirt, handle the piece only by the edges or the top; it may be wise to use a Kimwipe while handling the clean metal.
   CAUTION: Acetone is flammable. After cleaning the aluminum, throw the acetone-soaked towel in the trash, and be sure your area is free from acetone before proceeding.

3. Take time now to examine the Al strip and record your observations. You may want to collect some quantitative data about the strip also.

Q 1: Why do you think it is important to clean and minimize the handling of the aluminum strip?

4. Check that the power is off and the power supply is unplugged from the wall.

5. Connect the appropriate alligator clip to the sheet above the electrolyte level to the NEGATIVE terminal of the power supply.

6. Connect the cleaned 1 cm x 8 cm aluminum piece at its top with an alligator clip and suspend the Al strip in the electrolyte solution so that only the bottom 2/3 of the strip is immersed. Be sure to keep the clip and the top of the strip out of the electrolyte. It might help if you put a ring stand and clamp nearby to hold the wire and red clip away from the sides. Connect the free end of the wire, opposite the clip, into the POSITIVE side of the power supply (if it has not been done already.)

7. Turn on the power supply (which should have its voltage pre-set at 10 volts) and allow the cell to run 5.0 minutes. Write down your observations of the cell while it is running. When done, turn the voltage off, and disconnect the power supply at the wall **before** handling the alligator clips.

8. **Remembering the caution about use of sulfuric acid,** carefully remove the aluminum piece from the sulfuric acid solution, disconnect the alligator clip, rinse the aluminum piece with distilled water, and pat dry with a Kimwipe. Observe the piece carefully and collect any additional data that may be needed, but remember to handle it minimally and then only with Kimwipes.

9. Write a brief explanation of you observations of any changes that may have occurred with the aluminum strip. Record this in your lab notebook and show it and the supporting data to your instructor.

PART II: To Dye For ...
You have several tasks in this part of the investigation.

TASK 1
The first task is to determine what effect an organic dye has on the anodized strip of aluminum. "Anodized" means it has served as the anode in the electrochemical process as undertaken in Part I. Rit brand dye, normally used to dye cotton and other fibers, will be used. Three strips of aluminum that have been previously anodized will be available from your instructor. Each strip in the set will have been anodized for either 5, 10, or 15 minutes.

Preparation of a Dye Bath
1. Measure 20 mL of the old liquid dye into a 150-mL beaker and add 80 mL distilled water to the dye. Stir and heat to about 60 to 70°C. If you are using new dye, use 20 mL of vinegar and 60 mL of water for your solution.

CAUTION: Remember to handle the anodized strips only minimally and then with Kimwipes.

2. Place the three aluminum anodized test-strips from your instructor AND your treated aluminum anode from Part I into the dye bath and allow them to remain there for 5–10 minutes. Keep the bath warm (60–70°C) but well below boiling.

3. Remove the aluminum strips from the bath and rinse them with distilled water. Observe the results.

4. Measure the conductivity of your strip with the appropriate meter.

Q 2: Which strip do you suspect of being treated as the anode for the shortest time? Which strip do you suspect of being treated as the anode for the longest time? Does a strip appear to have been treated as the anode for 5 minutes? Why or why not? Does the dye process affect the conductivity of the strip? Why or why not?

TASK 2
Using a new, clean strip of aluminum, prepare a strip that will match the color intensity of one of the three anodized strips previously provided to you by your instructor. Take the time to identify and control all variables that will affect this process. Keep a written record of your actions.

Once you have a strip you believe fits the criteria, show this to your instructor and also provide details of the procedure you followed to accomplish this task. Be sure to include this in your written record as well.

TASK 3

The third task you will perform is to determine the effect of heating the anodized metal before dying. Heat a beaker of distilled water to boiling. Obtain an anodized aluminum strip from your instructor. Place it in a boiling water bath for 5 to 10 minutes. Remove this strip from the boiling water bath and put it in the warm dye bath for 5 to 10 minutes. Remove, rinse the strip with distilled water, and observe the result.

Q 3: What you think occurred in the boiling water bath? What implication might this have for determining the temperature of the dye bath?

Q 4: Do you think the dyed strips should be treated in the boiling water bath? Why or why not?

TASK 4

The final task of Part II is to test the relative reactivity of the anodized and un-anodized aluminum strips in dilute hydrochloric acid. (2.5 M HCl is suggested.) Clearly note (1) the procedure you followed, (2) how each type of strip reacted in the hydrochloric acid, and (3) your explanation of any differences that you observed.

CAUTION: Do not drop the aluminum strips into the drain when cleaning up from this investigation. Put them into the solid waste container.

PART III: THE SCENARIO

Anodized aluminum cottage art has just caught the fancy of an artist friend of yours and you've been asked how difficult anodizing aluminum is and what type of dyes can be used.

By now you probably have an idea as to which dyed aluminum pieces you find most attractive and/or durable so you might decide that you want to fashion an ornament or jewelry-type item of aluminum.

Use your accumulated knowledge of the anodizing process in designing and treating your art object. Pay particular attention to the size of your piece and length of time the piece is anodized.

Keep a written description of what you did to make the art object and what the results are. The observable quality of your finished product will be a measure of your understanding of the chemistry of the process of anodizing aluminum.

CAUTION: The edges of the cut aluminum can be sharp and cause cuts so generally an article like a tree ornament or wall or desk decoration might be preferred to a pin that must be handled carefully and fastened to clothing.

ALUMINUM ADVENTURE
INSTRUCTOR NOTES

Time Required
The approximate time to complete this activity in the lab, including a pre-lab discussion is about $3^1/_2$ hours. (Our students ran out of time in one lab period and only completed Parts I and II.) Part III can be completed the following week or omitted entirely.

Group Size
Students can work individually or in pairs. (We chose for students to work in pairs.) There may be a limited number of electrochemical cells available for the students to work with. If this is the case, students can split into two groups so that half the class can do the first part, while the second half begins Part II.

Materials Needed
per group or student:
- electrochemical cell (a 400-mL beaker containing 3 M H_2SO_4 and an aluminum anode—see below for preparation)
- ringstand and clamp (to hold the aluminum strip in the center of the electrochemical cell)
- power supply (must be able to supply 10 volts)
- 2 wires with alligator clips on one end (to connect to the power supply)
- three 250-mL beakers
- 15-mm test tube
- hot plate (for dye bath and sealing bath)
- forceps
- paper towels or Kimwipes
- 2–3 untreated 1 cm x 8 cm aluminum strips
- aluminum strip anodized for 5 minutes
- aluminum strip anodized for 10 minutes
- aluminum strip anodized for 15 minutes
- piece of aluminum (for the art object; can be of varying sizes)
- magnifying lens
- conductivity tester

per class (20 students/20 set-ups):
- 6 L of 3 M sulfuric acid: To make 1 L, carefully add 167 mL of concentrated H_2SO_4 to 833 mL of distilled water. (If there is more than one section of lab, the same sulfuric acid can be reused.)
- 100 mL of 2.5 M hydrochloric acid: To 79 mL of distilled water carefully add 21 mL of concentrated HCl.
- 500 mL of acetone

Preparation of Electrochemical Cell

1. Cut a piece of aluminum sheet approximately 10 cm x 20 cm. You can use the same sheets for preparation of the strips and the students' experiment. However, before the lab begins, clean the sheet so that a fresh surface of aluminum is exposed. You can use sand paper to expose a new aluminum surface, then rinse with water and dry.
2. Clean dirt and grime off the aluminum sheet with acetone and rub dry with a paper towel.
3. Roll the sheet into a cylinder and place the aluminum cylinder into a 400-mL beaker. DO NOT CONNECT THE ALUMINUM TO THE POWER SUPPLY.
4. Add slightly less than 300 mL of 3 M H_2SO_4 to the beaker.

The cell is now ready for student use.

Preparation of Aluminum Strips

1. Cut the aluminum into 1 cm x 8 cm strips. Each student or group will need at least 2 untreated aluminum strips, one strip to anodize for 5 minutes and one strip to match the color intensity of the instructor's strip. We found that several groups needed 1 or 2 additional strips (no treatment) to match the color intensity of the instructor's strip.
2. Clean the strips that you will anodize for the students with acetone and rub dry with a paper towel.
3. Each group will need three aluminum strips that have been anodized for 5, 10, and 15 minutes respectively. If you are using more than one power supply to prepare the anodized strips, be sure to use a voltmeter to set the output of the power supply to 10 volts. The same output percentage may result in a different output voltage on each power supply. (The power supplies should be set to give an output of 10 volts for the students as well.) DO NOT MAKE ANY CONNECTIONS AT THIS TIME.
4. Pour slightly less than 300 mL of 3 M H_2SO_4 into the 400-mL beaker.
5. *Before turning the power supply on,* connect the negative terminal of the supply to the aluminum sheet (the anode) and the positive terminal to the aluminum strip (the cathode).
6. After you have checked that the connections are correct and the power supply is set to have an output of 10 volts, you can turn the power supply on and begin timed anodization of the aluminum strips. If you find that the aluminum strip is breaking off and falling into the solution, the output voltage of the power supply is set too high. Check the output voltage with a voltmeter and adjust it to 10 volts.

Notes

✔ Some dyes work better than others. When Rit brand dye was used a variation was found among the different colors. Some colors were accepted by the anodized strips much better than others. We suggest you test your dyes before students begin the laboratory experiment.

✔ Students may want different size pieces of aluminum to make their art object. You may want to cut pieces approximately 6 cm x 6 cm. Students can then cut this piece to any shape. You can also give students scrap pieces. Warn students to be careful when cutting the aluminum; the edges of the metal are very sharp.

Safety, Disposal, and Special Handling
Review the Material Safety Data Sheet (MSDS) of any chemical used in the experiment for information regarding safety and handling. Dispose of any waste according to your local ordinances.

Caution the students about the power supply. It is a good idea for you to check each student's setup before turning the power supply on. Remind them to follow instructions carefully. Be sure that students unplug the power supply from the outlet BEFORE they disconnect the aluminum pieces.

Points to Cover in Pre-lab
- If students have not been introduced to electrochemistry, you will need to define the following concepts:
  - ➢ electrochemistry—the study of chemical changes that are caused by or produce electricity
  - ➢ oxidation—a loss of electrons, also referred to as the combination of an element with oxygen (For example, the oxidation of iron to form iron oxide, commonly referred to as rust. Here, iron is oxidized and combines with oxygen.)
  - ➢ reduction—a gain of electrons
  - ➢ anode—the electrode where oxidation takes place
  - ➢ cathode—the electrode where reduction takes place
- Define anodize—to put a protective oxide coating onto a metal by an electrolytic process in which the metal serves as the anode. This method is used often in modern architecture to give a bronze or another appealing color to aluminum.
- Warn students to be careful with the power supplies. Also, students should have you check their setup before they turn the power supply on.
- Instruct students not to adjust any knobs on the power supply; you should have already set the voltage to 10 volts.

Likely Play-Out of Lab
In Part I, students record their observations of an untreated aluminum strip. The aluminum strip is shiny and has a smooth surface. Next, students anodize the aluminum strip for 5 minutes and formulate a hypothesis to explain their observations. The aluminum is being oxidized to aluminum ions which then react with oxygen (available in the solution because of the reduction of water at the cathode—students should observe bubbles formed at the cathode). Aluminum oxide is formed on the surface of the aluminum strip and the strip becomes dull and gray.

In Part II, students place three strips that have been anodized by the instructor in a dye bath along with their strip from Part I. The strips accept the dye. The strip that was anodized for 15 minutes will have the most intense coloring. The strip anodized for 5 minutes will have the least intense coloring.

Students then have to match the color intensity of one of the strips that you provided and that they have just dyed. Most students will choose to match the strip that was anodized for 5

minutes since it matches their anodized strip (which they also anodized for 5 minutes) and they don't have to guess the time of anodization. However, students do prefer the more intensely colored strips and some choose to match that color. The main variable that affects the color intensity of the strip that students identify is the time of anodization. Other variables they may mention include the amount of dye in the dye bath, the temperature of the dye bath, and the initial cleanliness of the aluminum strip.

Next, students place another anodized aluminum strip into a boiling water bath before placing the strip in the dye bath. This strip does not dye as well as the previous strips. This is because the aluminum oxide has been sealed by the boiling water bath and cannot accept any dye molecules.

Students then test the reactivity of an anodized and un-anodized aluminum strip in dilute hydrochloric acid. The anodized aluminum strip will not react in the acid since it has a protective coating of aluminum oxide on its surface. (Aluminum metal also reacts in air to form aluminum oxide. This chemically resistant oxide coating is the reason aluminum can be used to make airplanes, cooking utensils, etc.) The un-anodized strip of aluminum reacts with hydrochloric acid, and bubbles are produced due to the formation of hydrogen gas.

Finally, students can design a piece of fashion jewelry using the information they have learned from the experiment.

Possible Answers to Questions

Q1: Why do you think it is important to clean and minimize the handling of the aluminum strip?

A clean aluminum surface is needed since the aluminum atoms on the surface of the strip are oxidized to Al^{3+} in the electrochemical cell. Students need to minimize the handling of the aluminum strip since oil from fingers covers the aluminum atoms on the surface, preventing the atoms from being oxidized.

Q2: Which strip do you suspect of being treated as the anode for the shortest time? Which strip do you suspect of being treated as the anode for the longest time? Does a strip appear to have been treated as the anode for 5 minutes? Why or why not?

The strip that appeared to have the thinnest coating (darkest in appearance, most closely resembling the untreated aluminum strip) was anodized for 5 minutes. The strip with the whitest and dullest look (the "coating" looks thicker) is the strip that was treated for the longest time, 15 minutes. One of the treated strips seems to have been anodized for 5 minutes since it has the same amount of dullness and closely resembles the strip students anodized for 5 minutes.

Q3: What do you think occurred in the boiling water bath? What implication might this have for determining the temperature of the dye bath?

When the anodized strip was placed in the boiling water bath without any dye present, the strip is sealed and will no longer accept dye. For this reason, it is important that the dye bath temperature be maintained well below boiling (thus the suggested temperature of 60–70°C).

Q4: Do you think the dyed strips should be treated in the boiling water bath? Why or why not?

Yes, the dyed strip should be treated in the boiling water bath. The boiling water bath seals the dye and prevents the strip from accepting any more dye. In addition, the dye cannot wear off of the strip since it is sealed into the aluminum metal.