

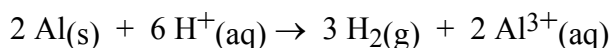
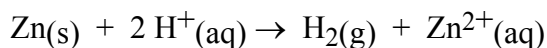
# Analysis of an Alloy

## Introduction

In this experiment, you will use the reaction of hydrochloric acid with an aluminum/zinc alloy to determine the percentage of aluminum in the alloy. This is an example of the mixture problems you have previously studied. You will determine the mass of an alloy sample, determine the moles of hydrogen gas the sample produces and then use this information to determine the composition of the alloy. Like most things, this is easier said than done. You should already be familiar with the calculations necessary for this experiment. If you are not, go back and review them before you try to do the experiment.

In most of the examples you have worked you were given the mass of the sample and an indication of the number of moles involved. However, in the process of collecting the hydrogen gas released in the reaction of hydrochloric acid with the alloy you will also collect some water vapor which must be accounted for. The following paragraphs tell you how to deal with these additional complications as well as give you a brief outline of the calculations involved.

The composition of the Al/Zn alloy can be determined by the amount of hydrogen gas produced from the reaction and the mass of the alloy. The reactions of interest are:



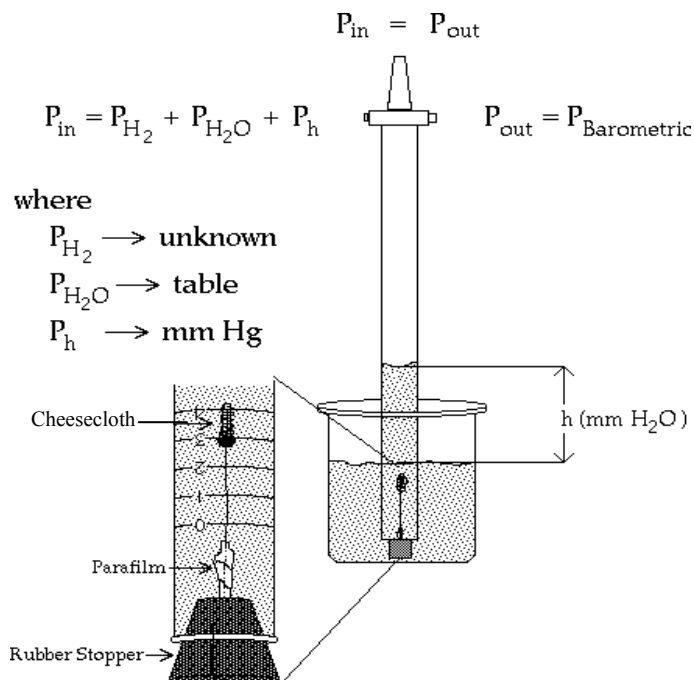
Note: for each mole of reacted zinc, one mole of hydrogen gas is produced. For every two moles of aluminum that reacts, three moles of hydrogen are generated.

These reactions are performed in an inverted buret so that the generated hydrogen gas can be collected and the volume measured as shown in Figure 1.

With the measured volume of hydrogen gas, the total pressure of the gas inside the buret, and the room temperature, the moles of hydrogen gas produced from the sample can be calculated using the Ideal Gas Law:

$$P_{(\text{H}_2)}V = n_{(\text{H}_2)}RT$$

The volume of hydrogen gas is measured from the buret, and the temperature reading can be easily obtained. The problem is to determine the pressure of the dry hydrogen gas. Note that the pressure inside the buret must be equal to the pressure outside the buret (Figure 1). The pressure outside of the buret is the atmospheric or barometric pressure, which is determined by reading the barometer mounted on the wall in the laboratory room. The inner pressure is (1) the total gas pressure ( $\text{H}_2$  plus  $\text{H}_2\text{O}$ ) and (2) the pressure exerted by the column of water extending above the water level in the beaker, as is illustrated in Figure 1. The total gas pressure is the sum of the partial pressure of the hydrogen produced and of the vapor pressure of water, which is temperature dependent and can be found in Table 1. The pressure exerted by this column of water is related to its density. For example, as you know, a barometric pressure of 1 atm is equal to 760 mm Hg. This means that if mercury were placed in your inverted buret, the barometric pressure would support a column of mercury 760 mm high. This would be the  $h$  in Figure 1. If the fluid were one-half as dense as mercury, then the column would be twice as high. If you used water, which has a density of 1 g/mL as compared to mercury, which has a density of 13.6 g/mL, then the column supported by 1 atm would be  $13.6 \times 760$  mm. This is about 32 feet!



**Figure 1: Diagram of Apparatus**

To calculate  $P_{H_2}$  you need to take into consideration both the vapor pressure of water and the height difference,  $h$ . Consider the following equations:

$$P_{in} = P_{out}$$

This must be true or the water would be moving in the buret! You know the value of  $P_{out}$ , as this is the barometric pressure, which you can read off the barometer mounted in the laboratory room. **Please do not touch any of the buttons on the barometer.** It follows, therefore, that you know the value of  $P_{in}$ . Furthermore,

$$P_{in} = P_{H_2} + P_{H_2O} + P_h$$

In the above equation,  $P_{in}$  is known and  $P_{H_2O}$  may be obtained from Table 1. To solve for  $P_{H_2}$  you just have to figure out  $h$  and express it in a consistent set of units! You can measure  $h$  with a ruler, but that gives you **mm of water** and you want **mm of mercury**. To convert, you simply need to use the density of water (1 g/mL) and mercury (13.6 g/mL) to find  $P_h$  in units of mm of mercury. As a hint, keep in mind that the height of the liquid is inversely related to the density. The details of this calculation are left to you.

**Table 1: Vapor Pressure of Water at Different Temperatures**

Temperature (°C)	Vapor Pressure (mm Hg)	Temperature (°C)	Vapor Pressure (mm Hg)
15	12.8	23	21.1
16	13.6	24	22.4
17	14.5	25	23.8
18	15.5	26	25.2
19	16.5	27	26.7
20	17.5	28	28.3
21	18.6	29	30.0
22	19.8	30	31.8

You now can solve for  $P_{H_2}$ , and you can measure the temperature and the volume that the hydrogen occupies. This allows you to solve the Ideal Gas Law for the total moles of hydrogen produced by the Al/Zn alloy. Now you should be in familiar territory. If you know the mass of the sample you can let:

$$x = \text{grams of Al}$$

$$\text{then, } N - x = \text{grams of Zn.}$$

where  $N$  is the mass of the sample. You can relate these mass expressions to the moles of hydrogen produced using the balanced chemical equations. That is, the moles of hydrogen produced by Al plus the moles of hydrogen produced by Zn must be equal to the total moles of hydrogen produced. Now you have the data to solve the mixture problem for the amount of aluminum in the alloy.

**Safety:** Wear gloves when using concentrated HCl. The hydrogen gas produced is flammable; NO FLAMES in the lab. Wear your goggles.

### Procedure

Work individually on this experiment.

Since you are collecting a gas for the first time, you are being given more detailed instructions than you have been in previous experiments. Do not allow yourself to fall into the trap of following the recipe without understanding how the ingredients work together. You are going to have to use these techniques again in another experiment.

1. As a preparation for the actual experiment we will need to determine the volume between the 50 mL mark and the top of the stopcock in your buret. This volume varies with each buret and so it is important to do this step for your buret before beginning the experiment. Use the buret clamp on your lab bench to hold your 50 mL buret in place. To calibrate your buret, first fill the buret with water above the 50 mL mark and let it drain to near the 50 mL mark. Now, **with the buret tip filled with water**, slowly drain the water out into a 25 mL graduated cylinder until the water level reaches the top of the buret stopcock. Measure and record the amount of water. Subtract the volume of water that was above the 50 mL mark to find the volume of the unmarked portion of the buret. This unmarked volume of the buret will be occupied by hydrogen gas when the buret is inverted. Thus this volume will be added to the marked volume in order to determine the total volume of hydrogen gas produced. (This will make more sense to you after you have completed the first trial.)

Question A: Carefully explain why we need to measure the volume of the buret between the 50 mL mark and the top of the stopcock.

2. Obtain your unknown from your TA. Weigh samples that are between 35 and 45 milligrams. These samples are very small, so be careful not to lose them. The accurate measurement of the mass of the unknown is very critical. Therefore, be as accurate as possible when measuring the unknown. Use weighing paper and make sure the balance pan is clean before weighing. Wait for the balance to stop fluctuating

to obtain an accurate reading. Do not touch the samples with your hands to prevent them being contaminated by oils on your fingers.

3. Wrap the first sample **in a 1 cm square** piece of cheesecloth. Use one end of a piece of copper wire to fasten the bundle. It is important that you use as little cheese cloth as possible, use a single layer and cut away any excess.
4. Leave about four inches of the wire to hook through a size 00 rubber stopper with a single hole. You want the length of the wire long enough so that when you place the stopper in the buret, the capsule will be between the third and fourth milliliter markings.
5. Place 250 mL of deionized water in an 800 or 400 mL beaker and place it beneath the buret. If you are using a 400 mL beaker, make sure that you can get the buret into the water in the beaker without losing any water from the buret. If you can not do this add about 50 ml of water to the beaker. From the bottle in the hood, measure out 20 mL of concentrated hydrochloric acid into a graduated cylinder. **Caution: concentrated HCl causes severe burns. Wear gloves when working with concentrated acids.** Pour the acid into the buret and slowly fill the buret up to the rim with deionized water. As you perform the next step be sure that the end of the buret is not pointing toward you or anyone else. VERY CAREFULLY use your water bottle, to gently pour the water so that it drips along the buret wall in order to reduce the mixing of the acid and water. It is CRITICAL that you SLOWLY pour the dI water ALONG THE BURET WALL. If water is added to concentrated acid too quickly it will produce enough heat to boil over and spray acid. Make sure the buret is as full as possible.
6. Place the unknown into the buret and close the opening with the rubber stopper. It is crucial to make sure that no air bubbles are trapped in the buret. Unclamp the buret and invert it by placing a gloved finger over the hole of the rubber stopper and turning the buret upside down. Submerge the stopper and the bottom few centimeters of the buret into the beaker of water before releasing the finger. Place the buret back into the buret clamp. Concentrated HCl is more dense than water, so the acid will diffuse down, react with the alloy, and produce hydrogen gas. With the production of  $H_2$ , the liquid is then forced out of the buret. Once the reaction is complete, an equilibrium will be established between the pressure inside the buret and the atmospheric pressure outside the buret.
7. As you watch the reaction, take notice of any bubbles being released through the hole of the stopper. If there is a large flow of gas coming out, you must redo the experiment because of hydrogen gas escaping. This problem is generally due to either the metal sample being too close to the stopper.
8. Once the entire sample is dissolved, tap the buret a few times to dislodge the gas bubbles from the wall of the buret and from the copper wire. Let the buret cool for a few minutes to reach room temperature. Determine the volume of hydrogen gas produced by reading the milliliter markings, subtracting this measurement from 50, and adding the volume of the calibrated, unmarked portion of your buret. Measure the height difference of the water level in the buret from the water surface in the beaker using the ruler provided.

9. To dismantle the apparatus first open the stopcock to drain out the liquid in the buret. Rinse the buret, beaker, wire, and stopper with deionized water, and repeat the procedure with two more samples of your unknown. If the reaction was slow then, then increase the amount of acid used to 30 mL, and if the reaction was too fast then decrease the volume to 10 mL.

Do not attempt to run this experiment with multiple burets. There are only enough burets for you to do one sample at a time. If you try to run multiple samples you will cause a shortage of burets.

10. Measure the room temperature. The barometric pressure will be posted on the wall opposite the stockroom dispensary window. You will need this pressure in the calculations for this lab.
11. Now calculate the percentage of aluminum in the sample. This is not an easy calculation and so you should do as much of it as possible during the laboratory period. Also, make arrangements to meet with your TA to go over these calculations during office hours or during discussion if you are still having trouble with them.

Write-up. Calculate an average, a standard deviation, a 90% confidence limit, and a relative deviation for the percentage of aluminum in your unknown sample.