EXPERIMENT 1

"…GRAPPLE THEM TO THY SOUL WITH HOOPS OF STEEL…" DETERMINATION OF THE COMPOSITION OF AN ALLOY

Techniques

- analytical weighing
- gas collection over water

MSDS available for

- zinc, Zn
- magnesium, Mg
- alloys
- hydrochloric acid, HCl (aq)

Principles

- mole ratios
- Dalton's law of partial pressures
- stoichiometry
- reactions of acids and metals
- ideal gas law

Recommended Advanced Reading

- **Chapter 3,4 in Silberberg, Lavieri, and Venkateswaran, 1st CE, McGraw-Hill, 2013.**

INTRODUCTION

The beginning

In this experiment you will be working with gas laws and stoichiometry. In today's session you will work with your partner to:

- determine the uncalibrated volume of a eudiometer tube
- calculate the number of moles of a pure metal reacted with an acid based on the amount of hydrogen collected during the reaction
- compare the calculated number of moles with the number of moles of metal determined by the mass of metal used
- use the same technique to determine the percentage of two metals in an unknown alloy

This is a general overview of what you will be accomplishing in this experiment.

EXPERIMENT 1: *Determination of the Composition of an Alloy*

Introduction

"Aluminum is quite a soft metal, but when a small amount of copper is mixed in, the alloy duralumin is produced, which is strong enough to be used in aircraft frames!"

Reactions of Metals and Acids

When a metal reacts with an acid, hydrogen gas is liberated. The amount of hydrogen liberated is directly proportional to the amount of metal that was reacted. The ***stoichiometry*** of the reaction indicates the relation between the amount (mol) of metal reacted and the amount (mol) of hydrogen produced.

For instance, the reaction between calcium and hydrochloric acid is:

$$Ca\,(s) + 2\,HCl\,(aq) \rightarrow CaCl_2\,(aq) + H_2\,(g)\uparrow \qquad [1]$$

Since this reaction is taking place in an aqueous medium, the ***net ionic equation*** may be written as,

$$Ca\,(s) + 2\,H^+\,(aq) \rightarrow Ca^{2+}\,(aq) + H_2\,(g)\uparrow \qquad [2]$$

where Cl^- is a spectator ion and is cancelled on both sides. From both equations [1] and [2], it can be seen that for every mole of calcium reacted, one mole of hydrogen gas is liberated.

On the other hand, in the reaction between chromium and hydrochloric acid, the net ionic equation can be written as

$$Cr\,(s) + 3\,H^+\,(aq) \rightarrow Cr^{3+}\,(aq) + 3/2\ H_2\,(g)\uparrow \qquad [3]$$

and it can be seen that for every mole of chromium reacted, **three-halves** of a mole of hydrogen gas are liberated.

If the number of moles of hydrogen released in a reaction between a metal and an acid is measured, the amount of metal reacted can be determined using the above stoichiometry. This is a simple task when there is only one metal involved. What happens when there are two metals involved, as in the case of an alloy?

Reaction of an Alloy and Acid

When there are two metals involved, the amount of hydrogen liberated must be equal to the amount of hydrogen that each metal would liberate individually. Thus, if an alloy consists of 1 g of iron and 1 g of chromium, the amount of hydrogen that **should** be liberated is the amount of hydrogen that is released by 1 g of iron **plus** the amount of hydrogen that is released by 1 g of chromium. The first can be determined from the stoichiometry of the reaction of iron and the acid and the second can be determined from the stoichiometry of the reaction of chromium and the acid.

The alloy you will be using in this experiment is made of zinc and aluminum. The net ionic equations for the two reactions involved are

$$Zn\,(s) + 2\,H^+\,(aq) \rightarrow Zn^{2+}\,(aq) + H_2\,(g)\uparrow \qquad [4]$$
$$Al\,(s) + 3\,H^+\,(aq) \rightarrow Al^{3+}\,(aq) + 3/2\,H_2\,(g)\uparrow \qquad [5]$$

From these equations, the stoichiometry indicates that 1 mole of Zn produces 1 mole of hydrogen gas, and 1 mole of Al produces 3/2 mole of hydrogen gas. Thus the total amount of hydrogen gas is equal to the amount of hydrogen gas produced from the zinc plus the amount of hydrogen gas produced from the aluminum, or

$$n_{\text{hydrogen total}} = n_{\text{hydrogen, zinc}} + n_{\text{hydrogen, aluminum}} \qquad [6]$$

From equations [4] and [5] we can see that the number of moles of hydrogen gas produced from the zinc is the same as the number of moles of zinc. However, the number of moles of hydrogen gas produced from the aluminum is 3/2 times the number of moles of aluminum (more hydrogen gas is produced per mole of aluminum). We can now rewrite equation [6] as

$$n_{\text{hydrogen total}} = n_{\text{zinc}} + 3/2\ n_{\text{aluminum}} \qquad [7]$$

$$= \frac{\text{mass of zinc}}{\text{molar mass of zinc}} + \frac{3}{2}\,\frac{\text{mass of aluminum}}{\text{molar mass of aluminum}}$$

The equation for the amount of hydrogen has thus reduced to an equation in two unknowns: the mass of zinc and the mass of aluminum. Unfortunately, it is impossible to solve a system of one equation in two unknowns! There is, however, one other equation relating these two unknowns, and that is

$$m_{\text{total of alloy}} = m_{\text{zinc in alloy}} + m_{\text{aluminum in alloy}} \qquad [8]$$

We now have two equations in two unknowns. This system of equations can be solved by any method you choose.

Concept of the Experiment

In this experiment, there are two main parts and one you may have to do. First, you will verify if you have a calibrated or uncalibrated eudiometer. If it is already calibrated, you can proceed to the experiment; if not, you will need to determine the volume of the uncalibrated portion of the eudiometer tube. Second, you will determine the mass of a sample of a pure metal, react it with a given volume of concentrated hydrochloric acid, and trap the hydrogen gas liberated by the reaction. **Remember!!** You are trapping the hydrogen gas over water and you must subtract the water vapour pressure from the total pressure to obtain the pressure of the dry hydrogen. You will then use the Ideal Gas Law to determine the amount (mol) of hydrogen gas and the stoichiometry of the balanced equation to determine the amount (mol) of metal that reacted. You will then compare this value to the amount (mol) of metal that was actually reacted (after all, you know the mass!). Finally, you will repeat part two of the experiment with an alloy sample, and use equations [7] and [8] to determine the percentage of each metal in the alloy sample.

One last point is that the hydrogen gas is being collected over water. On the surface of the water, the pressure inside the tube must equal the pressure outside the tube. The pressure inside the tube is

$$P_{inside} = P_{hydrogen} + P_{water\ vapour} \quad [9]$$

and the pressure outside the tube is

$$P_{outside} = P_{atmospheric} - P_{water\ column} \quad [10]$$

Combining equations [9] and [10], we can calculate the hydrogen pressure.

$$P_{hydrogen} = P_{atmospheric} - P_{water\ column} - P_{water\ vapour} \quad [11]$$

(TTD) Things to Do

- See the video for this experiment and complete all the prelab exercises before coming to the laboratory.
- Determine the volume of the uncalibrated portion of the eudiometer tube.
- Measure the volume and calculate the pressure of hydrogen gas obtained by reaction of a known mass of a known metal with concentrated hydrochloric acid.
- Use the same method with a sample of an alloy and determine the percentages of the two metals that compose the alloy.
- Use safe laboratory procedures at all times.

Safety Precautions

1. **Wear approved eye protection at all times**.
2. The acid solution supplied is concentrated. Concentrated acids are **corrosive**. Dilute acids are also corrosive. Be careful!!
3. Although you are producing only a small quantity of hydrogen, do not breathe it!

PROCEDURE

Equipment and chemicals needed

Chemicals	*Equipment*
concentrated HCl	600 mL or 1000 mL beaker
magnesium metal	wash bottle
zinc metal	eudiometer tube
alloy sample	10 mL graduated cylinder
sample holder and vial	analytical balance

Determination of the Composition of an Alloy

Starting the experiment

Calibrating the Tip of the Eudiometer Tube (if you have a eudiometer tube with an empty space on top of the ZERO Mark and starts at 0.00 mL). OTHERWISE, proceed to the next section!

1. Place between 8 and 10 mL of distilled water in the 10 mL graduated cylinder. Record the exact volume reading on your raw data sheet.

2. Transfer the distilled water into the eudiometer tube. Read and record the volume at the meniscus of the water column in the eudiometer tube.

3. Determine the volume of the uncalibrated tip of the eudiometer by difference. Repeat this measurement at least twice and record all data.

Moving on!

Reaction of a Pure Metal with Acid

4. Obtain a sample of pure metal from your demonstrator and record its identity.

5. Use Table 2, provided at the end of this section, to determine the quantity of metal that must be used. Obtain ROUGHLY the correct mass (using the pan balance). Polish each piece of metal thoroughly using sand paper. Without touching the metal with your fingers, wipe each piece well to remove the shavings. Use the analytical balance to obtain a sample with the specified mass.

6. Place the metal sample in the sample holder and place the sample holder in the vial. Half fill a 600 mL (or 1000 mL) beaker with distilled water and immerse the sample holder and vial in the beaker (you can fill the sample holder and vial with distilled water using a wash bottle first). BE CAREFUL!

Ensure that the entire sample remains in the sample holder. See the setup in Figure 1. (There should be NO air anywhere!)

7. Use Table 2 to determine the volume of acid required for the sample of metal that you have been assigned.

8. Place the concentrated acid (12 M HCl) in the eudiometer.

9. Use a wash bottle to add distilled water to the eudiometer tube. Add the water in such a way as to form a layer on top of the acid (try to have as little dilution as possible!) Fill the tube to the brim leaving a bubble on top.

10. Place your forefinger on the top of the filled tube and quickly invert the tube. Place the mouth of the tube under the water in the beaker before removing your finger. Quickly place the mouth of the eudiometer tube over the sample holder so that the sample vial surrounds the eudiometer tube that surrounds the sample holder. Ensure that no air bubbles are trapped in the tube. Clamp the tube in place.

11. If the metal, once it starts to react, begins to float upward in the eudiometer tube, tap the tube gently to ensure that the metal remains in the acid solution. If it sticks to the glass wall of the tube, you will have to repeat the experiment.

12. Once the formation of hydrogen has visibly ceased, tap the tube to ensure that no bits of metal remain to react.

13. Record the volume of gas collected. Remember to include the volume of the tip of the tube (if necessary). Record the temperature of the water.

14. Use a ruler to determine the height, in centimetres, of the column of water (from the surface of the water in the eudiometer tube to the surface of the water in the beaker).

15. Repeat this procedure at least once with the same metal.

16. Calculate the number of moles of hydrogen formed and metal used.

Almost there!

Determining the Composition of an Alloy

17. Obtain a sample of alloy from your demonstrator and record its unknown number.

18. Consult the table at the end to determine what mass of sample must be used and what volume of acid is needed to react with the sample. Use the analytical balance to measure the exact mass of sample you use.

19. Repeat steps 6 to 15 with two different masses of your alloy sample.

Finishing the Experiment

20. Record the atmospheric pressure and the room temperature.

21. Calculate the percentage of each metal in the alloy.

22. Remember to get your raw data, written in PEN, signed by your TA and to attach this raw data to your report in order to receive a grade!

Cleaning Up!

23. Dispose of any remaining metal or alloy (unused in the experiment) by wrapping it in brown paper and throwing it in the trash bin.

24. All solutions can be poured down the drain and flushed with plenty of water. Rinse all beakers, the eudiometer tube and replace them in the drawer. Wipe the bench clean and dispose of paper towels in the trash bin.

<u>Calculations:</u>

Pure metal

1. Calculate the volume of the uncalibrated tip of the eudiometer tube by subtracting the volume you read on the eudiometer tube from the volume you read on the graduated cylinder. Then determine the actual volume of hydrogen gas collected.

2. Calculate the pressure of the water column using the density of water, the height of the column and the acceleration due to gravity (P = dgh).

3. Use Equation [11] to calculate the pressure of the hydrogen gas (find the pressure of the water vapour in Table 1).

4. Use the ideal gas law to determine the amount (mol) of hydrogen liberated by the pure metal (DON'T forget to have consistent UNITS!).

5. Calculate the amount (mol) of hydrogen that should be formed by the amount (mol) of metal that you reacted.

6. Determine the percent yield of hydrogen formed (actual yield/theoretical yield). (For this calculation, you may choose to use mass actual/mass theoretical or moles actual/moles theoretical). Does this number depend on the mass of metal you chose to react?

Alloy

7. Determine the volume, pressure and amount (mol) of hydrogen gas for the alloy.

8. Use a combination of Equations [7] and [8] to determine the masses of zinc and aluminum in your alloy. (Express the mass of zinc in terms of the mass of aluminum and the total mass of the alloy).

9. Determine the percentage composition of the alloy.

10. Does the percentage composition depend on the mass of alloy you measured? Do you expect it to be the same for both samples?

Points to Ponder

- How close were the two values calculated for amount (mol) of metal used? Is your answer reasonable?
- What are the sources of error inherent in the experiment? How does each source of error contribute to the result (ie. does it increase or decrease the value of the amount (mol) or percentage of metal?)

Lab Report

- *Refer to page iii in the FYI section and the lab report section in experiment 1.*
- Complete all the calculations, as outlined in the procedural section, and **don't forget to state all results with the correct number of significant figures!!**

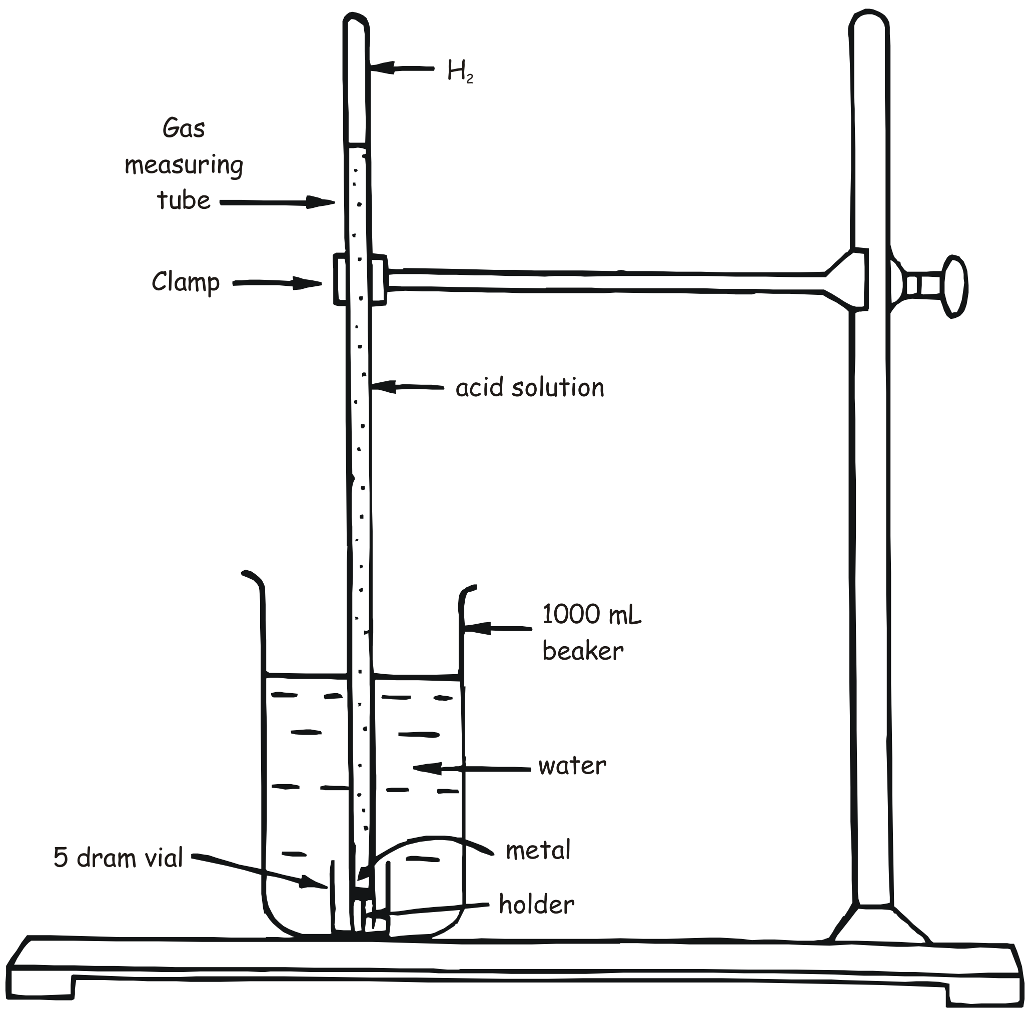
Table 1. Water Vapour Pressure

Temperature °C	Pressure kPa	Temperature °C	Pressure kPa
15	1.71	23	2.81
16	1.82	24	2.98
17	1.94	25	3.17
18	2.06	26	3.36
19	2.20	27	3.57
20	2.34	28	3.78
21	2.49	29	4.00
22	2.64	30	4.24

Table 2. Volume of Acid Required for Specific Masses of Metal and/or Alloy

Substance	Mass of Substance	Volume of HCl Needed
-	mg	mL
Aluminum, Al	12 - 15	22-23
Magnesium, Mg	20 - 30	10
Zinc, Zn	40 - 60	20
Alloy (Zn and Al)	about 40	10

Figure 1. Sample Setup of Apparatus for the Determination of the Composition of an Alloy



Rubric for Correction of Experiment 1

Tables (6)		Recopied neatly from raw data and complete for 2 trials each	Recopied neatly from raw data but 1 trial missing;	Recopied neatly from raw data but 2 trials missing;	Recopied neatly from raw data but not complete	Not recopied from raw data but complete	Not recopied and not complete or not included
Observations (4)			Complete for all parts	Partial for all parts	Complete for one part	Partial for one part	None
Calculations (7) (metal)	All calculations shown and neatly explained	All calculations shown	Most calculation shown neatly	Most calculation shown	Some calculation shown neatly	Some calculation shown	Calculations incomplete
Calculations (alloy) (6)		All calculations shown and neatly explained	All calculations shown	Most calculation shown neatly	Most calculation shown	Some calculation shown neatly	Calculations incomplete
Discussion (5)		Clear explanation of results based on observation and data; reasonable explanation for errors; comparison between trials; comparison between metal and alloy; ties to theory clearly shown	Explanation of results; some connection to observation/data; explanation of errors; some comparisons; some ties to theory	Explanations are vague; connections to observation/data unclear; comparisons or ties to theories poorly explained	A few important points missing	Many important points missing	no discussion
Conclusion (2)					One statement per part. Results clearly stated.	Results somewhat evident.	Results not evident/missing.
Total	30						