Electrolytic Determination of Equivalent Mass

Objectives

The objective of this laboratory is to use electrolysis to determine the equivalent mass of an unknown metal.

Background

In a previous lab you determined the equivalent mass (EM) of an unknown acid by titrating it with a standardized NaOH solution. For that procedure the equivalent mass of the acid was defined as the mass of acid that produced one mole of H^+:

$$\text{EM of acid} = \frac{\text{mass of acid}}{\text{moles of } H^+}$$

In the context of a redox reaction the equivalent mass of a metal can be defined as the mass of metal that will produce one mole of electrons:

$$\text{EM of metal} = \frac{\text{mass of metal}}{\text{moles of electrons}}$$

The redox reaction will take place in an electrolytic cell, which consists of two electrodes submerged in an electrolyte solution. The electrodes are connected to an external power source, thus completing the circuit. Electrons will be transferred from the anode of the power source to the cathode of the electrolytic cell where the reduction of hydrogen will occur:

$$2 \, H^+ \, (aq) \; + \; 2 \, e^- \; \rightarrow \; H_2 \, (g)$$

Oxidation of the unknown metal will take place at the anode of the electrolytic cell and these electrons will be transferred to the cathode of the external power supply:

$$M \, (s) \; \rightarrow \; M^{n+} \, (aq) \; + \; n \, e^-$$
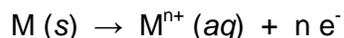
This process is represented by Figure 1 on the following page.
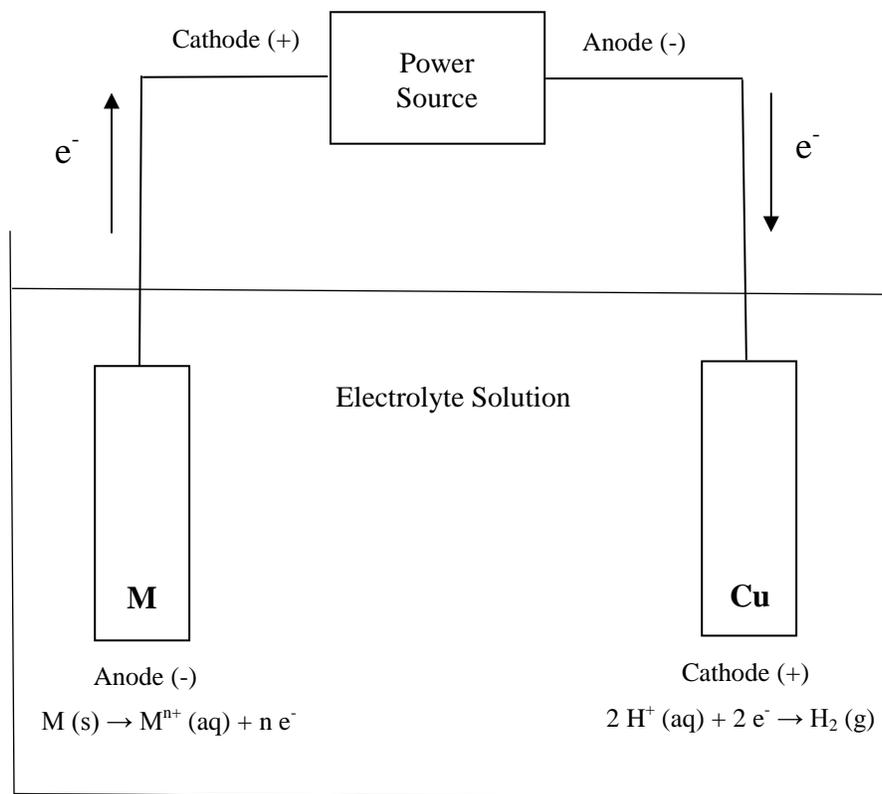
Figure 1

Procedure

Chemicals

Unknown metal, conducting electrolyte solution of 0.1 M $HC_2H_3O_2$ in 0.5 M Na_2SO_4 (*aq*), acetone

Equipment

5-volt power supply, sand paper, eudiometer tube (with cork/stopper), buret stand and clamp, 250-mL beaker, 100-mL graduated cylinder, thermometer, barometer, electrolysis kit

Safety and Waste Disposal

① The 0.1 M $HC_2H_3O_2$ in 0.5 M Na_2SO_4 must be disposed of in the hazardous waste container!

② Acetone is flammable! It should be kept far away from any open flame and the bottle should be closed when not in use.

③ The power supply should be turned off when not in use.

Instructions

1. Obtain a piece of the unknown metal from your instructor. Lightly sand the metal to remove all surface impurities. Rinse the metal first with water and then with acetone, and let the acetone evaporate. Once dry, weigh the metal on the analytical balance and record this mass to the nearest 0.001 g.

2. Figure 2 below illustrates the overall set-up of equipment for this experiment. Please refer to this figure while assembling your own equipment in the next steps.
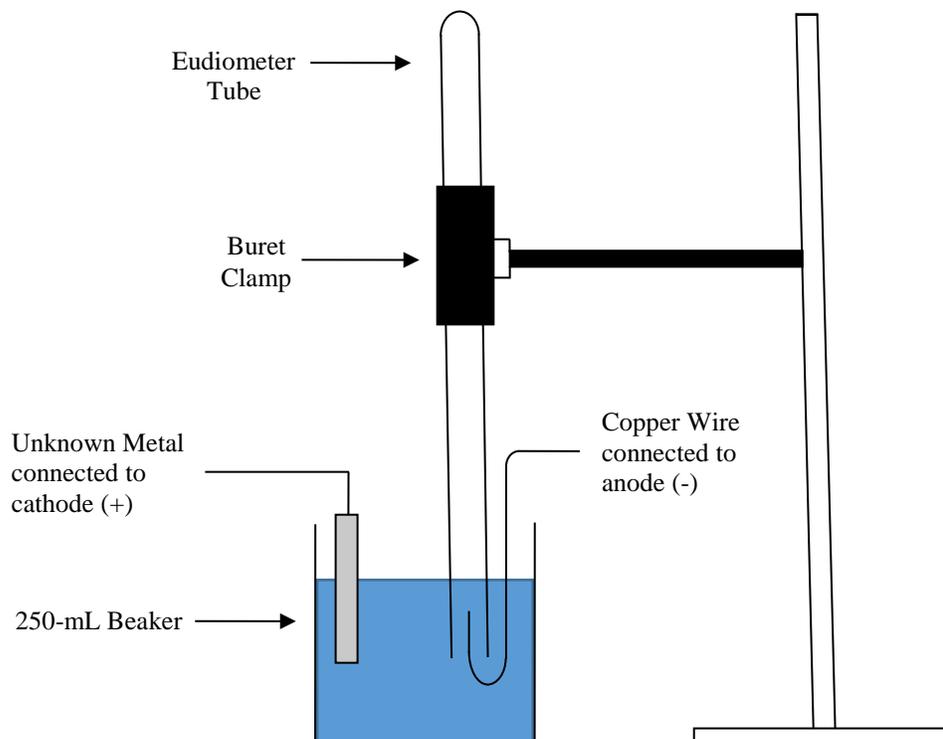

Figure 2

3. Obtain two insulated wires from your electrolysis kit, each with a banana plug on one side and an alligator clip on the other side. Using one of the insulated wires, connect the alligator clip to the unknown metal and place the metal in the 250-mL beaker. The alligator clip should be used to secure the metal to the side of the beaker.

4. Now take the second insulated wire and connect the alligator clip to the supplied copper wire (in kit). Place the copper wire in the 250-mL beaker **on the opposite side from the unknown metal**. The alligator clip should be used to secure the copper wire to the side of the beaker.

5. Add 100 mL of 0.1 M $HC_2H_3O_2$ in 0.5 M Na_2SO_4 (the conducting electrolyte solution) to the 250-mL beaker. The ends of the unknown metal and the copper wire should be immersed in this conducting solution, however, **make sure that the alligator clips do not come into contact with the solution.**

6. Fill a eudiometer tube to the brim with the conducting electrolyte solution. Place a cork or a stopper in the end of the tube, invert it, and carefully place it in the beaker below the surface of the solution.

7. Remove the stopper and position the tube so that the exposed part of the copper wire is completely inside the tube. **Make sure that the mouth of the tube does not break the surface of the solution.** Secure the eudiometer tube in place using a buret clamp and a buret stand.

8. Use the banana plug attached to the first insulated wire to connect the unknown metal to the cathode (positive, red) of the 5-volt power supply. **Make sure that the power supply is off at this point.** Then use the banana plug attached to the second insulated wire to connect the copper wire to the anode (negative, black) of the 5-volt power supply.

9. Now turn on the power supply. You should see bubbles of hydrogen gas being generated on the exposed surface of the copper wire and rising up inside the eudiometer tube. Collect between 45 mL and 50 mL of hydrogen gas, then **turn off the power source**. Record the volume on the eudiometer tube to the nearest 0.01 mL.

10. Carefully remove the eudiometer tube from the solution in the beaker. Re-fill it to the brim with conducting electrolyte solution, cork it, and place it back into the solution in the beaker (as in Step 6). Re-insert the copper wire inside the tube and clamp the tube in place (as in Step 7).

11. Turn on the power supply and again collect between 45 mL and 50 mL of hydrogen gas. Then turn off the power source, and record the volume on the eudiometer tube to the nearest 0.01 mL.

12. Measure and record the temperature of the electrolyte solution and the atmospheric pressure.

13. Remove the unknown metal from the beaker and disconnect the attached clips and wires. Again, rinse the metal with water followed by acetone. Let the acetone evaporate, then weigh the dry metal on the analytical balance to the nearest 0.001 g.

14. Remove the eudiometer tube from the beaker, and disassemble all your equipment. Discard the electrolyte solution in the supplied waste container. The eudiometer tube must be rinsed with water before it is returned. The copper wire must be rinsed with water and acetone before it is returned.

Name: _____   Date: _____

Lab Partner: _____   Lab Section: _____

Lab Report: Electrolytic Determination of Equivalent Mass

Experimental Data:

Mass of metal before electrolysis	
Eudiometer volume after 1st electrolysis	
Eudiometer volume after 2nd electrolysis	
Total volume of hydrogen collected	
Mass of metal after 1st and 2nd electrolysis	
Atmospheric Pressure	
Temperature of electrolyte solution	
Vapor Pressure of H_2O at above temperature*	

Obtain this value through an online source. Include in your answer where you obtain the reference from.

Calculations:

Calculate the equivalent mass of your unknown metal. Show your complete work below (and on the back of this page as needed).