A Thesis Presented to

The Faculty of Alfred University

Electrolysis Lab for General Chemistry Students

By

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Introductory Statement:

College is a place where students are given the opportunity to explore classes and expand their horizons. Classes fall into a variety of categories which allows for students to have a wide range of classroom experiences before graduation. Alfred University is no exception to this. Even with small class sizes, Alfred allows for its students to discover new potential that they wouldn't have found before. In having different schools on one campus, many students are required to take a natural science course with a lab if you are in the College of Liberal Arts and Sciences, as well as the College of Business. First year engineering students are also required to complete General Chemistry 105 in the fall and Chemistry 106 in the spring. With three to four sections of General Chemistry each semester at varying times, many students will take advantage of this and attempt to successfully pass.

The General Chemistry courses include not only lecture three times a week, but also one day of lab demo and a two hour lab. Even though lab has no credits, it is factored in as 20% of the overall grade. Chemistry lab allows for students to take what they have learned in lecture and apply it in a hands on environment. Another benefit would be that the majority of the time, work is completed in partners or groups. By having students work together, this allows them to develop skills in communication as well as learning how to work with others. In doing so, chemistry labs are preparing students for jobs in the future as most employers are looking for these skills regardless of the field of work chosen. Chemistry lab offers potential future scientists a chance to see what actual lab life can be. It is crucial to understand that not all experiments work perfectly, since science is not perfect. Lab is an excellent time for students to grow both individually and alongside their peers.

In being a senior chemistry major, I have personally enjoyed labs and feel that there are many skills students can learn in a lab environment. For this reason, I have decided to focus my thesis on finding a new lab that would be more beneficial than the ones that are currently used in the department. While the old labs don't have any prominent issues, change or addition of a new lab will benefit the program and future students. This is a tall task as there are thousands of different general chemistry labs published by universities all around the world. However, it is important that a new lab would have purpose as well as a direct correlation to the topics that are taught in lecture. In searching the web for labs that would be appropriate, I found an electrolysis lab that would be an appropriate future experiment.

Electrolysis is a topic that involves redox chemistry. All lecture sections of General Chemistry 106 are required to cover redox chemistry. When one mentions redox, they are referring to a chemical reaction that involves the movement of electrons. Typically, there are two different species in the system being studied. One of these will gain electrons as the reaction proceeds while the other species will lose elections. If a species gains electrons, it undergoes the process of reduction and if a species loses electrons, it undergoes oxidation. This is where the term redox comes from (reduction and oxidation).

Redox is typically taught during the first couple weeks of April which would require it to be one of the later labs in the semester. Once redox is covered in class, all students will have the necessary skills to not only perform this lab but to also succeed in doing any accompanying calculations. It is important for lab practices to not just spoon feed students the correct answers but rather allow for guidance in understanding the math behind the science. In performing the electrolysis lab, it allows for more thinking and actual understanding of what is occurring rather than just spitting the correct answers out and forgetting what was taught.

With regards to the actual lab itself, it is not difficult. The chemistry behind it is rather straight forward but some may find difficulties if there is no prior experience with electrical circuits. The schematic that is given along with the lab gives the students a direction to follow. When performing this lab, students will visibly see the changes in the chemical reaction as the solution will turn blue and bubbles will form. The change from clear to blue represents the copper going from solid copper to copper $^{+2}$(aq). The bubbles represent the creation of hydrogen gas which will be measured by a buret. This is an important aspect of lab because it allows the students to work on writing down clear, meaningful observations. With obvious color changes and the formation of bubbles, the electrolysis lab allows for a good lab experience in taking observations.

Students will also work on reading burets. This has been a troublesome area for many students, even after the laboratory practical in early March. Students have difficulties remembering whether to read burets top to bottom or bottom to top. They also experience issues with significant figures. With this lab, there is another difficult aspect with reading glassware. The buret needed for this lab is upside down which means students have to read it backwards in order to get the correct reading. In planning on running the lab twice with a partner, this allows the students to ask questions not only of the professor and lab assistants but from their peers as well. Sometimes the best way to reinforce a topic is to teach it to someone else. This lab gives the students plenty of ways to strength their lab practices that they should have previously learned.

This lab has been performed several different times by several different students including myself, an upper level chemistry student, and four general chemistry students. It is important to note that the percent yield for this experiment has been a rather difficult aspect. For

the trials done by myself and the upper level student, the percent yield for either hydrogen or copper was over 100%. Trials were changed to try to improve upon the percent yield. The electrodes were sanded and different ways of drying were also explored as well. The hope was to obtain realistic percent yields (below 100%). With the general chemistry trials, their second trial was successful in getting both the hydrogen and the copper percent yield below 100%. This was successful due to allowing the electrode to air dry and not dry manually. In not further removing any copper on accident, the percent yield is right where it needs to be.

Currently, there are no experiences with electrolysis in a laboratory setting in the chemistry department. The coursework though includes redox to be covered in several classes including in depth in upper level chemistry classes but never hands on experience. By adding this lab to the curriculum, there will be numerous students who have experience with electrolysis at an early level. This can allow for further exploration in electrolysis. It provides more confidence when setting up circuits as well as critically thinking about how to move forward when something doesn't work. By having a lab in electrolysis, it shows students another topic that chemistry can offer.

The importance of this lab is not only to benefit the future students of Alfred University but to benefit the chemistry department. In recent years, the department has been working with first year students in testing out new labs that could potentially become a part of the lab course for incoming students. As previously mentioned, this lab has been performed by students of many different skill sets. It is one thing to have senior chemistry students to be able to perform this lab but if first years with very limited knowledge can perform the lab successfully, then there is a strong case for it to be a new lab for the department. In writing this thesis, essentially the hard parts of trial and error, writing out the procedure so that it follows suit of previous labs, and

working out calculations in order to apply to second semester of chemistry are completed. With more trials of this lab, it has the potential to be put in effect as early as next spring (2019).

The last but potentially the most important reason to do this new lab would be because of its cost effectiveness. With the renovation of the general chemistry labs this upcoming summer (2018), the department can place a power supply in every bench. This can be covered by the renovation fees so no extra money needs to be spent. All of the students have their own drawers full of necessary supplies such as beakers. The wires for this lab are inexpensive and only so many are needed to have enough so that each lab can work as partners. The copper foil needed for this lab can be ordered on a mass scale and is reusable. The electrodes can be reused as well. Finally, the sulfuric acid is a common acid that is in stock for other experiments and would simply require larger quantities to be ordered to account for this lab. The solution can be reused for both trials of the experiment as well. Therefore, this lab is very low cost which is helpful to a department that is not in possession of a disposable budget.

All in all, this thesis provides a potential future lab for the Alfred University chemistry department. With all of the work done on this experiment, I see no reason why the department wouldn't highly consider this added this lab to the Chemistry 106 lesson plan. With this electrolysis lab, students will learn about circuits, redox chemistry, and reinforce prior skills. It is a type of lab that has not been previously seen by most students which allows the growth of understanding electrolysis. By expanding what is done in laboratory is not only beneficial for the department but for the students as well. Adding this lab will better the department and strengthen the knowledge that general chemistry students can take with them.

Historical Background:

Electrolysis is a mixture of both electricity and chemistry. It is allowing for the measurement of different electrical quantities such as current, potential, charge, or a relationship to chemical parameters (Wang). The history of electrolysis dates back to the early 1800's. It is important to note that even before the 1800's, people such as Benjamin Franklin were studying electricity and static current. It wasn't until Alessandro Volta whom had been successful in producing a continuous current, that a scientist gets credit for discovering of electrolysis. He was the one who invented the "voltaic pile", which is considered the first modern electrical battery. In doing so, he set the way for the study of chemical production of electricity as well as what the possible effects of electricity was on chemicals.

The reasoning behind Volta's creation of the first electric battery arises out of a rather strange story. Volta had built the battery to prove that Luigi Galvani was incorrect in stating that animals can produce electricity. Galvani had used two different metals that were connected by a frog's leg (conductor) in order to produce a current. He believed that this is how muscles worked throughout the body. Volta, with the voltaic pile, successfully disproved Galvani as he had used zinc and silver disks that were connected by brine-soaked cardboard. The current simply passes through the materials and didn't originate from it.

In disproving Galvani, this opened the way for studying other properties of certain metals. Volta, like many other scientists, believed that there was a way to rank metals and other species according to their electrical strengths. He is credited with publishing one of the first electromotive series. These were similar to affinity tables that chemists had been working on years prior. In doing so, this had allowed for another scientist, Humphry Davy, to confirm that

this electrical theory of chemical affinities wasn't just random and was very important ("The History of Electrochemistry: From Volta to Edison.").

Davy can be argued as being one of the backbone designers of the electrochemical experiments. He determined that the production of electricity depends on the chemical reaction that occurred, not just the type of metals. In using electricity, Davy was able to successfully separate compounds which led to the discovery of new elements such as sodium, potassium, calcium, magnesium, boron and barium ("Humphry Davy"). Davy argued in 1806 that since electrical current can overpower the normal force that holds the elements together that the force therefore must be electrical in nature. This helped set the foundation of current day electricity.

Following the discoveries made by Davy, his student Michael Faraday continued his research. Faraday is a common and well-known name in the sciences. Faraday was successful in proving the relationship between electricity and magnetism. Faraday is accredited with inventing the first electric motor in 1821 as well as the first generator in 1831. In 1834, he published his works that included two laws of electrochemistry ("The History of Electrochemistry: From Volta to Edison."). Faraday's Law states that the amount of a substance produced at each electrode is directly proportional to the quantity of charge flow through the cell ("Faraday's Law"). This law is directly used in this lab to determine how much product can be obtained by passing a certain amount of current through a system. The more common name for this process is electrolysis.

Scientists continued to explore the possibilities of electrolysis. With this, electroplating became very popular. Up until the mid-1800's, it was very uncommon to have silverware if you were not a part of a wealthy family. Electroplating occurs when one metal is coated onto another one, most commonly for decorative purposes or to prevent corrosion of a metal. It allows for inexpensive metals to be used for the majority of the product and then apply different metals on

the outside to account for appearance, protection, and other desired properties for a product ("Electroplating.")The process was inexpensive as just a coat of silver was placed onto silverware give the illusion of being completely made out of expensive metals. This continued on to other items such as bumpers on cars, computer parts, baby shoes, etc. and is still used today.

With respect to industry, electrolysis was a key component in the 1890's as it was used to found some of the largest chemical companies that are still in business today. Herbert H. Dow was the first scientist to use electrolysis to remove bromine from Michigan brine wells. He then turned around and sold the bromine to medicine and photography companies. Dow also used the process to extract chorine. This process led to The Dow Chemical Company in 1897. This company expanded its processes to extract other chemicals from brine to use them in manufacturing processes such as dyes and plastics. The company continued on after Dow's death to extract chemicals from seawater and use the chemicals for profit such as creating lightweight metal alloys for airplanes in WWII ("The History of Electrochemistry: From Volta to Edison.").

Scientists continued to explore the possibilities with electrolysis and discovered that it can help prevent corrosion. Corrosion is essentially the breaking down of metals, stones or other materials. The most common kind of corrosion is from electrochemical reactions. The best example of this is an old rusting car. The rust that is on the car is from the metal being oxidized. Rusting will occur over time but also with repeated exposure to moisture as well as oxygen. Bridges follow similar suit and with the high costs of repairs, often times another metal will be electroplated on so that it is oxidized first instead of the bridge. This deals with the standard reduction table (Figure 1).

Standard Reduction Potentials in Aqueous Solution at 25°C

Reduction Half-Reaction		$E°$ (V)
$F_2(g) + 2\ e^-$	$\rightarrow 2\ F^-(aq)$	+2.87
$H_2O_2(aq) + 2\ H_3O^+(aq) + 2\ e^-$	$\rightarrow 4\ H_2O(\ell)$	+1.77
$PbO_2(s) + SO_4^{2-}(aq) + 4\ H_3O^+(aq) + 2\ e^-$	$\rightarrow PbSO_4(s) + 6\ H_2O(\ell)$	+1.685
$MnO_4^-(aq) + 8\ H_3O^+(aq) + 5\ e^-$	$\rightarrow Mn^{2+}(aq) + 12\ H_2O(\ell)$	+1.52
$Au^{3+}(aq) + 3\ e^-$	$\rightarrow Au(s)$	+1.50
$Cl_2(g) + 2\ e^-$	$\rightarrow 2\ Cl^-(aq)$	+1.360
$Cr_2O_7^{2-}(aq) + 14\ H_3O^+(aq) + 6\ e^-$	$\rightarrow 2\ Cr^{3+}(aq) + 21\ H_2O(\ell)$	+1.33
$O_2(g) + 4\ H_3O^+(aq) + 4\ e^-$	$\rightarrow 6\ H_2O(\ell)$	+1.229
$Br_2(\ell) + 2\ e^-$	$\rightarrow 2\ Br^-(aq)$	+1.08
$NO_3^-(aq) + 4\ H_3O^+(aq) + 3\ e^-$	$\rightarrow NO(g) + 6\ H_2O(\ell)$	+0.96
$OCl^-(aq) + H_2O(\ell) + 2\ e^-$	$\rightarrow Cl^-(aq) + 2\ OH^-(aq)$	+0.89
$Hg^{2+}(aq) + 2\ e^-$	$\rightarrow Hg(\ell)$	+0.855
$Ag^+(aq) + e^-$	$\rightarrow Ag(s)$	+0.80
$Hg_2^{2+}(aq) + 2\ e^-$	$\rightarrow 2\ Hg(\ell)$	+0.789
$Fe^{3+}(aq) + e^-$	$\rightarrow Fe^{2+}(aq)$	+0.771
$I_2(s) + 2\ e^-$	$\rightarrow 2\ I^-(aq)$	+0.535
$O_2(g) + 2\ H_2O(\ell) + 4\ e^-$	$\rightarrow 4\ OH^-(aq)$	+0.40
$Cu^{2+}(aq) + 2\ e^-$	$\rightarrow Cu(s)$	+0.337
$Sn^{4+}(aq) + 2\ e^-$	$\rightarrow Sn^{2+}(aq)$	+0.15
$2\ H_3O^+(aq) + 2\ e^-$	$\rightarrow H_2(g) + 2\ H_2O(\ell)$	0.00
$Sn^{2+}(aq) + 2\ e^-$	$\rightarrow Sn(s)$	−0.14
$Ni^{2+}(aq) + 2\ e^-$	$\rightarrow Ni(s)$	−0.25
$V^{3+}(aq) + e^-$	$\rightarrow V^{2+}(aq)$	−0.255
$PbSO_4(s) + 2\ e^-$	$\rightarrow Pb(s) + SO_4^{2-}(aq)$	−0.356
$Cd^{2+}(aq) + 2\ e^-$	$\rightarrow Cd(s)$	−0.40
$Fe^{2+}(aq) + 2\ e^-$	$\rightarrow Fe(s)$	−0.44
$Zn^{2+}(aq) + 2\ e^-$	$\rightarrow Zn(s)$	−0.763
$2\ H_2O(\ell) + 2\ e^-$	$\rightarrow H_2(g) + 2\ OH^-(aq)$	−0.8277
$Al^{3+}(aq) + 3\ e^-$	$\rightarrow Al(s)$	−1.66
$Mg^{2+}(aq) + 2\ e^-$	$\rightarrow Mg(s)$	−2.37
$Na^+(aq) + e^-$	$\rightarrow Na(s)$	−2.714
$K^+(aq) + e^-$	$\rightarrow K(s)$	−2.925
$Li^+(aq) + e^-$	$\rightarrow Li(s)$	−3.045

Figure 1- Standard Reduction Potentials for given elements (Wordpress.)

The lower the metal is on the list, the more likely it will oxidize first. This indicates that it is a stronger reducing agent. This saves companies money in the long run because it is cheaper to replace a metal like magnesium as opposed to an entire steel bridge.

Corrosion is just one of the applications that electrolysis can help slow down. In the 1980's and early 1990's there were several new developments that were made thanks to studies of electrolysis. Some of these include the creation of ultra-micro electrodes, the design of tailored interfaces and molecular monolayers, the coupling of biological components and

electrochemical transducers, and the development of ultra-trace voltammetry techniques and microfabrication of molecular devices or efficient flow detectors (Wang).

Chemical Background:

With all of the given history of electrolysis, it is important to further explore exactly what is taking place during this type of chemical reaction. There are two different types of electrochemical cells. One is electrolytic (which is seen in the experiment) and it consumes electricity from an external source. The other is galvanic which produces electrical current. There needs to be a measurement of cell potential which is the difference in potential across the electrodes of a cell. This measureable difference in potential will indicate whether a current is passing through the cell or not (Bard).

The overall chemical reaction taking place in a cell is made up of two independent half reactions which describe the real chemical changes at the two electrodes. Each half reaction responds to the interfacial potential difference at the corresponding electrodes. Most of the time one is interested in only one of these reactions. If this is the case, it is called the working electrode. The electrochemical process that is taking place in this laboratory procedure occurs at the electrode-solution interface (Wang).

Electrodes can also vary based on what is needed for the experiment. When it comes to solid electrodes, they have considerable analytical interest due to their extended anodic potential windows. The most common working electrodes are carbon, platinum, and gold. Silver, nickel, and copper can also be used. There is also a dependency on response of the surface state of the electrode. The use of such electrodes require precise electrode pretreatment and polishing to obtain reproducible results. Solid electrodes present a heterogeneous surface with respect to the electrochemical activity. This leads to deviations of behavior expected from homogeneous

surfaces. For this reason, it is important to note that there can be a range of results in this experiment (Wang).

The objective of a controlled-potential electroanalytical experiment is to obtain a current response that is related to the concentration of the target analyte. A generic formula for this process is:

$$O + ne^- \Leftrightarrow R$$

Where O stands for the oxidized species, ne^- is the number of moles of electrons, and R is the reduced species. It is important to mention that this reaction is under Faradic current and will obey Faraday's Law, which states that the reaction of 1 mole of substance involves a charge of n times 96,487 coulombs. For the purpose of general chemistry, students are able to round to 96,500 coulombs and the calculations work the same.

In understanding the basics of the half-cell reaction, it is important to identify where all numbers compare. The internationally accepted primary reference is known as the Standard Hydrogen Electrode (SHE) which follows the following:

$$Pt/H_2\ (a=1)/H^+,\ (a=1,\ aqueous),\ E_{cell}= 0\ Volts\ (Wang)$$

This is also seen in Figure 1. We observe or control the potential of the working electrode with respect to the reference and that is equivalent to observing or controlling the energy of the electrons within the working electrode. It is also important to note that all predictions are made based on thermodynamic considerations and slow kinetics might prevent a reaction from occurring at a significant rate.

With relationship to the kinetics of the experiment, current also needs to be discussed. The current in which electrons cross the interface from metals to a species in a solution is termed a cathodic current. The pathway of an electrode reaction can be quite complicated and may even

take place in a sequence that involves several steps. The rate of the reaction is determined by the slowest step. Simple reactions involve only mass transport of the electroactive species to the electrode surface, electrons transfer across the interface, and then there is the transport of the product back to the bulk solution. Limitations are usually from either the mass transport of the reactant or the rate at which electrons can be transferred. Migration for electrochemical reactions is the movement of a charged body under the influence of an electric field (a gradient of electrical potential) (Bard).

The Experiment:

The electrochemical cell that is in question for this lab is constructed out of two different electrodes in a dilute solution of sulfuric acid. When the power supply is turned on, the flow of electrons will be from the anode to cathode. Oxidation will occur with the copper and will yield the following half reaction:

$$Cu\ (s) \rightarrow Cu^{+2} + 2e^{-}$$

Due to the oxidation occurring at the copper electrode, the copper will lose mass as the reaction proceeds and the solution that it is sitting in will turn blue due to the copper $^{+2}$(aq) ions. Given the above balanced equation, it is clear that for every one mole of copper solid, there are two moles of electrons being produced.

Since there is oxidation occurring, there must be reduction as well. The reduction will be the hydrogen gas which is visible in the buret. The half reaction of the hydrogen gas that is being produced is:

$$2H^{+} + 2e^{-} \rightarrow H_2\ (g)$$

In given the balanced equation, it is clear that for every one mole of hydrogen gas produced, there will be two moles of electrons. In figuring out the volume of hydrogen gas collected, as well as temperature and corrected pressure, one can use the ideal gas law,
PV = nRT, to determine the number of moles of hydrogen that are actually produced in this procedure.

With all of the information above, it is important to remember that the total charge that passes through a circuit can be determined by measuring the current for a certain amount of time. In this case, the DC current will be measured in units of amperes. An ampere is equivalent to 1 coulomb per second. For this experiment, you multiply current by the amount of time in seconds, to obtain the total charge in units of coulombs. Once the total charge is found, the Faraday constant of 96,500 coulombs in one mole of electrons to find the moles of electrons can be used that actually went through the system.

Lab Experiment:

BACKGROUND

Electrochemistry deals with the relation between electrical energy and chemical reactions. In an electrolytic cell, the chemical reactions happen when there is an external voltage applied. Since an external voltage is applied, work has to be done on the system for a reaction to occur. The voltage applied will move electrons. The copper electrode will lose mass as it is oxidizing ($Cu(s) \rightarrow Cu^{+2}(aq) + 2e^-$) and there will be the production of hydrogen gas ($2H^+ + 2e^- \rightarrow H_2(g)$) from the sulfuric acid which directly relates to the reduction. The overall balanced equation would therefore be $2H^+ + Cu(s) \rightarrow H_2(g) + Cu^{+2}(aq)$. By collecting this gas, we can follow simple calculations to determine the percent yield of H_2 (gas). In measuring the mass of the copper electrode, we can follow similar calculations to determine the percent yield of Cu.

FUNDAMENTAL LABORATORY TECHNIQUES

- Setting up a DC circuit
- Massing by difference and volume measurements (inverted buret)
- Solution Preparation

EXPECTED LEARNING OUTCOMES

- Determine balanced REDOX reaction
- Determine percent yield
- How to operate a DC power supply

CHEMICALS

Sulfuric Acid	H_2SO_4
Copper Foil	$Cu(s)$, $Cu^{+2}(aq)$

EQUIPMENT/MATERIALS

Burets	250 mL beaker	Balance	Graduated Cylinder
Copper Foil	DC Power Supply	Wires	Insulated electrode
Multimeter	Rubber Suction Bulb	Ring Stand	

OBJECTIVES

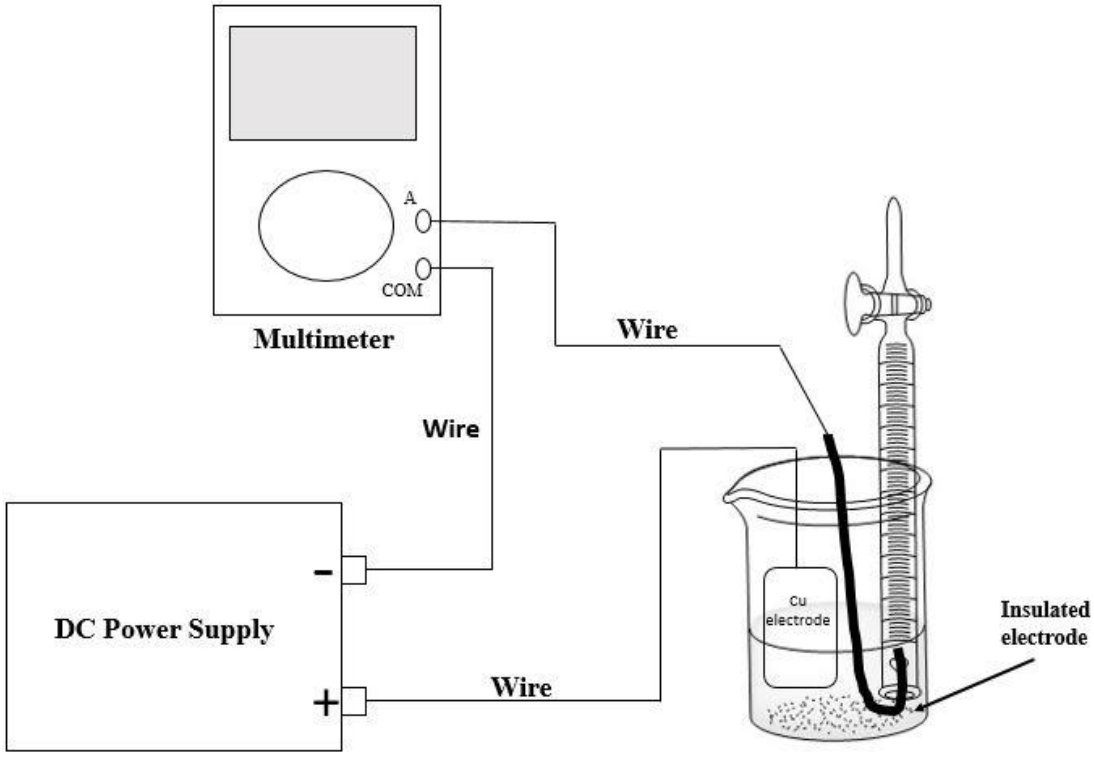
- Experimentally determine the percent yield of $H_2(g)$ and Cu(s) using an electrolytic cell

PROCEDURE

1. Work in pairs.
2. Prepare 150 mL of a 0.2 M solution of sulfuric acid in the 250 mL beaker
    a. What equation do you need to prepare a dilution?

b. Show your calculations in your lab notebook.

3. Use a ring stand and a buret clamp to fix a 50 mL buret upside down.
4. Place the insulated wire electrode into the inverted buret so that it sits about 3 mL up into the buret.
5. Put the inverted buret with the wire electrode inside of it into the 250 mL beaker that contains your 0.2 M sulfuric acid.
6. Be sure that the buret is **not** touching the bottom of the beaker.
7. Open the stopcock on the buret and use the rubber suction bulb to fill the buret with your solution up to the 50.00 mL mark.
8. Close the stopcock and ensure that the solution level remains constant in the buret.
9. Take your copper electrode and sand both sides.



10. Complete the remaining parts of the circuit as shown below:
11. Note: Do **not** let the wires that are connecting the electrode to the power source touch the solution.
12. Have the instructor or the TA check your set up before proceeding.
13. After your setup has been approved, test the circuit by turning on the power supply and turn the multimeter to the 10 amp scale. If the bubbles being formed are in the buret and the multimeter has a reading between 0.2 and 0.5 A, you can turn off the power supply.
14. Check to see where the meniscus is in the buret. If it has dropped below 47 mL, refill the buret by opening the stopcock and using the rubber suction bulb.
15. Record the initial reading on the buret in your lab notebook.
16. Unclip the copper electrode and remove it from the circuit.
17. Obtain the mass of the copper electrode and record it in your lab notebook.

18. Put the electrode back in the circuit by placing it back in solution and reattaching the wire.
19. Turn on the power supply (this is time zero) and start your stopwatch.
20. Record the current at time zero.
21. After 60 seconds has passed, record the current and the buret reading in the table into your lab notebook. It is recommended to start at the top of a new page to ensure room for all data points.

Time	Current (Amp)	Volume (mL)
Record	In	Notebook
Record	In	Notebook
Record	In	Notebook
Leave space	for more	entries

22. Continue recording current and volume measurements until your buret solution is 3-5 mL above the beaker solution.
23. Turn off power supply.
24. Measure the distance between where the solution is in the buret and where it lies in the beaker using a ruler and record this in your lab notebook in millimeters.
25. Obtain the lab temperature (in Kelvin) as well as the barometric pressure (in mm Hg) from your instructor or your TA.
26. Unclip your copper electrode avoiding contact with the acid solution and allow it to air dry.
27. Record the mass of the copper into your lab notebook.

28. Open the stopcock and allow the remaining solution to empty into your beaker.
29. Remove the insulated electrode from the buret and rinse with water.
30. Complete a second trial of this lab and be sure to record the necessary data.
31. Once you have completed your second trial, disassemble the remaining parts of your circuit.

CALCULATIONS

Determine the following calculations for each trial. Record in lab notebook, using proper units.

1. Determine the total charge of the reaction.

   Remember that 1 amp = $\frac{coulomb}{second}$

2. Determine the number of moles of electrons.

   Remember $\frac{1\ mole\ of\ electrons}{96500\ coulombs}$

3. Determine the theoretical moles of H_2 (g) using the balanced redox equation.

   Remember $2H^+ + 2e^- \rightarrow H_2$ (g)

4. Determine the theoretical moles of Cu (s) using the balanced redox equation.

   Remember: Cu (s) → Cu^{+2} (aq) $+2e^-$

5. Determine the volume of H_2 (g) collected.
6. Calculate the pressure of hydrostatic force using the formula below:

$$\text{Pressure}_{\text{hydrostatic}} = \frac{height\ of\ the\ liquid\ in\ the\ buret\ (mm)}{\frac{13.6\ mm\ of\ H20}{1\ mm\ of\ Hg}}$$

7. Using Table 1 in the Appendix, determine the pressure of H_2O based on the lab temperature.
8. Determine the pressure of H_2 (gas) using the formula below:

$$\text{Pressure}_{H2(g)} = \text{Pressure}_{\text{Barometer}} - \text{Pressure}_{\text{hydrostatic}} - \text{Pressure}_{\text{water}}$$

9. Convert the pressure of hydrogen gas from mm Hg to atm.
10. Determine the number of moles of hydrogen using PV=nRT and be sure to use the correct units given R=0.0821$\frac{L*atm}{mol*K}$.
11. Determine the moles of Cu oxidized in the experiment.
12. Calculate the percent yield of H_2 (g).
13. Calculate the percent yield of Cu (s).

WASTE DISPOSAL

All chemicals used in this experiment can be disposed of as hazardous waste and are collected into the appropriately labeled bottles located in the SAA.

CRITICAL THINKING

Answer the following questions in your lab notebook prior to writing your conclusion:

a. Does the shape of the copper electrode matter?
b. What factors affect the rate at which this reaction occurs?
c. Why do you need to use DC current and not AC?
d. Why does the solution turn blue as the experiment occurs?

CONCLUSION

In your own words describe how you met the objectives for this laboratory, give your results, and list sources of error that explain why your results might have deviated from what was expected. The critical thinking questions can be used to facilitate discussion in the conclusion.

REFERENCES

Adopted from Eastern Michigan University and edited by Melissa Marx.

Appendix:

Table 1- Vapor Pressure of Water at Various Temperatures

Temperature (˚C)	Pressure (mm Hg)	Temperature (˚C)	Pressure (mm Hg)
16	13.6	22	19.8
17	14.5	23	21.1
18	15.5	24	22.4
19	16.5	25	23.8
20	17.5	26	25.2
21	18.7	27	26.7

Safety/Chemical Hazards:

Copper Foil- May cause eye, skin, respiratory tract irritation.

Sulfuric Acid- Causes eye and skin burns. May be fatal if inhaled. Concentrated sulfuric acid reacts violently with water and many other substances under certain conditions.

Results:

The results from this lab come from myself, another senior chemistry student (Kaylin Brant), and four first years who are currently completing General Chemistry 106 this semester. All of the data that was collected was processed through Microsoft Excel with the given mathematical operations as functions in the sheet.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	261
0	0.29	48.3	0.0483		Number of moles of electrons	0.0027
60	0.29	47.6	0.0476		Expected number of moles of H2	0.00135
120	0.29	46.2	0.0462		Expected number of moles of Cu	0.00135
180	0.29	44.9	0.0449		Volume of gas collected	0.0365
240	0.29	42.7	0.0427		Pressure of hydrostatic (mm Hg)	4.77941
300	0.29	40.8	0.0408		Pressure of H20 (mm Hg)	18.7
360	0.29	38.9	0.0389		Pressure of H2 (mm Hg)	680.821
420	0.29	36.5	0.0365		Pressure of H2 (atm)	0.89582
480	0.29	33.5	0.0335		Number of moles of hydrogen	0.00135
540	0.29	31.54	0.03154		Moles of Cu oxidized	0.00183
600	0.29	29.63	0.02963		% yield of H2	100.081
660	0.29	25.8	0.0258		% yield of Cu	134.985
720	0.29	21.75	0.02175			
780	0.29	18.9	0.0189			
840	0.29	15.4	0.0154			
900	0.29	11.8	0.0118			
Average Current:	0.29				Difference between buret + beaker	65
					Pressure of Air	704.3
Initial Volume:	48.3		0.0483		R Value	0.0821
Final Volume:	11.8		0.0118		Temperature	294.261
Change in Volume	36.5		0.0365			
Initial Mass (grams)	1.687					
Final Mass (grams)	1.571					
Molar Mass of Copper	63.546					

Figure 2- Trial 1 of the experiment using old copper electrode. Performed by Melissa Marx

The first trial of the experiment was done using an old copper electrode that was previously used in a different electrochemistry experiment. It is important to note that for this trial, the current was constant throughout and was on the lower end of the suggested range. Due to this, it took 15 minutes for enough hydrogen gas to be collected. The percent yield for the hydrogen gas is right about where I was expecting it to be, a little above 100% as the buret system isn't perfect. However, the copper percent yield is over 100% which raises some red

flags. The main reason for this is due to the fact that the electrode was previously used and eroded. This trial allowed the realization that copper foil should be used instead to try to control the copper percent yield without harming the hydrogen.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	246.78
0	0.43	49.3	0.0493		Number of moles of electrons	0.002557
60	0.45	45.6	0.0456		Expected number of moles of H2	0.001279
120	0.45	41.1	0.0411		Expected number of moles of Cu	0.001279
180	0.45	37.5	0.0375		Volume of gas collected	0.0358
240	0.46	33.7	0.0337		Pressure of hydrostatic (mm Hg)	2.573529
300	0.46	29.6	0.0296		Pressure of H20 (mm Hg)	18.7
360	0.46	25.7	0.0257		Pressure of H2 (mm Hg)	685.2265
420	0.47	20.5	0.0205		Pressure of H2 (atm)	0.901614
480	0.47	17.7	0.0177		Number of moles of hydrogen	0.001339
540	0.47	13.5	0.0135		Moles of Cu oxidized	0.001275
					% yield of H2	104.7396
					% yield of Cu	99.68829
Average Current:	0.457				Difference between buret + beaker	35
					Pressure of Air	706.5
Initial Volume:	49.3		0.0493		R Value	0.0821
Final Volume:	13.5		0.0135		Temperature	293.56
Change in Volume	35.8		0.0358			
Initial Mass (grams)	4.575					
Final Mass (grams)	4.494					
Molar Mass of Copper	63.546					

Figure 3- Trial 2 of Electrolysis lab, performed by Kaylin Brant and Melissa Marx

The second trial of this lab was completed by senior chemistry major, Kaylin Brant, with assistance from myself. The main reason for having her try the experiment was to ensure that the written procedure was put in such a way that was understandable to people other than myself. The current was increased to run the experiment in less time but still yield the same amount of products. With this trial, we ran it using the copper foil electrode instead of the old copper piece. This helped to improve the yield of copper (99.69%) but now the hydrogen percentage is over 100. A reason for this could be due to the rounding errors that are from the pressure of water as

this was an estimate based on the temperature. This lab is also assuming that the temperature of the diluted solution is the same as the air which can cause error in the hydrogen gas calculations.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	255.8769
0	0.34	49.3	0.0493		Number of moles of electrons	0.002652
60	0.35	46.4	0.0464		Expected number of moles of H2	0.001326
120	0.35	43.2	0.0432		Expected number of moles of Cu	0.001326
180	0.35	40.1	0.0401		Volume of gas collected	0.0363
240	0.36	37.4	0.0374		Pressure of hydrostatic (mm Hg)	33.08824
300	0.35	33.8	0.0338		Pressure of H20 (mm Hg)	18.7
360	0.36	30.5	0.0305		Pressure of H2 (mm Hg)	653.9118
420	0.36	27.5	0.0275		Pressure of H2 (atm)	0.86041
480	0.36	24.4	0.0244		Number of moles of hydrogen	0.001291
540	0.36	21.5	0.0215		Moles of Cu oxidized	0.001401
600	0.36	18.4	0.0184		% yield of H2	97.37864
660	0.36	15.4	0.0154		% yield of Cu	105.6399
720	0.36	13	0.013			
Average Current:	0.355384615				Difference between buret + beaker	450
					Pressure of Air	705.7
Initial Volume:	49.3		0.0493		R Value	0.0821
Final Volume:	13		0.013		Temperature	294.667
Change in Volume	36.3		0.0363			
Initial Mass (grams)	4.455					
Final Mass (grams)	4.366					
Molar Mass of Copper	63.546					

Figure 4-Trial 3 of the experiment performed by Melissa Marx with sanded copper foil electrode

The third trial of this experiment was done with the copper foil that was previously used in trial two except this time it was sanded down to get any preliminary loose metal off. The current was assumed to be between the two previous trials only because that is where the initial reading was. It is important to note that no matter what current the system is ran at that it will produce approximately the same total charge. Given by the data above, the percent yield of the hydrogen gas was brought back below 100% but unfortunately the copper production was over 100%. The reasoning for this was due to improper drying of the copper electrode (used a Kim wipe to dry and took extra copper mass with it).

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	284.7
0	0.34	49.8	0.0498		Number of moles of electrons	0.00295
60	0.36	47.2	0.0472		Expected number of moles of H2	0.00148
120	0.36	44.2	0.0442		Expected number of moles of Cu	0.00148
180	0.35	41.1	0.0411		Volume of gas collected	0.0388
240	0.36	38.2	0.0382		Pressure of hydrostatic (mm Hg)	2.20588
300	0.35	35.3	0.0353		Pressure of H20 (mm Hg)	18.77
360	0.36	32.1	0.0321		Pressure of H2 (mm Hg)	684.524
420	0.37	29.2	0.0292		Pressure of H2 (atm)	0.90069
480	0.37	26.2	0.0262		Number of moles of hydrogen	0.00145
540	0.37	23.3	0.0233		Moles of Cu oxidized	0.00149
600	0.38	20.2	0.0202		% yield of H2	98.1491
660	0.38	17.2	0.0172		% yield of Cu	101.346
720	0.38	14.3	0.0143			
780	0.38	11	0.011			
Average Current:	0.365				Difference between buret + beaker	30
					Pressure of Air	705.5
Initial Volume:	49.8		0.0498		R Value	0.0821
Final Volume:	11		0.011		Temperature	294
Change in Volume	38.8		0.0388			
Initial Mass (grams)	4.338					
Final Mass (grams)	4.243					
Molar Mass of Copper	63.546					

Figure 5- Trial 4 of the experiment performed by Melissa Marx with sanded copper foil and sanded insulated electrode

The fourth trial of this experiment was performed similarly to the previous trial except the tip of the insulated electrode was also sanded in hope to improve results. The current was attempted to be constant at the same range as trial three. It should also be noted that if a trial is being repeated using the same diluted solution of sulfuric acid, there is more of a variance in the current. Once again, there was a reasonable percentage of hydrogen gas collected but the copper yield was over 100%. This trial was done in similar fashion as trial three with the electrode being dried by Kim Wipe which may have affected the results.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	285.6
0	0.3	49.7	0.0497		Number of moles of electrons	0.00296
60	0.31	47.5	0.0475		Expected number of moles of H2	0.00148
120	0.31	44.9	0.0449		Expected number of moles of Cu	0.00148
180	0.31	42.5	0.0425		Volume of gas collected	0.036
240	0.32	40	0.04		Pressure of hydrostatic (mm Hg)	1.838235
300	0.33	37.4	0.0374		Pressure of H20 (mm Hg)	18.77
360	0.34	34.8	0.0348		Pressure of H2 (mm Hg)	686.0918
420	0.35	32.3	0.0323		Pressure of H2 (atm)	0.902752
480	0.36	29.5	0.0295		Number of moles of hydrogen	0.001346
540	0.34	26.9	0.0269		Moles of Cu oxidized	0.001526
600	0.35	24.4	0.0244		% yield of H2	90.98708
660	0.36	21.8	0.0218		% yield of Cu	103.1532
720	0.37	19.1	0.0191			
780	0.37	16.5	0.0165			
840	0.38	13.7	0.0137			
Average Current:	0.34				Difference between buret + beaker	25
					Pressure of Air	706.7
Initial Volume:	49.7		0.0497		R Value	0.0821
Final Volume:	13.7		0.0137		Temperature	294
Change in Volume	36		0.036			
Initial Mass (grams)	1.638					
Final Mass (grams)	1.541					
Molar Mass of Copper	63.546					

Figure 6-Trial 5 of the experiment performed by Melissa Marx with old sanded copper electrode

The fifth trial of this lab was to test the hypothesis of trying to lower the percent of copper mass lost by sanding the old electrode. Unfortunately in using the old electrode, there was still over 100% yield in copper but a significant loss of hydrogen gas is noted from the other trials (approximately 10% wasn't recovered). After having completed this trial, I concluded that the old copper electrodes are not viable for this lab and that copper foil should definitely be used.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	266.75
0	0.39	49.9	0.0499		Number of moles of electrons	0.00276
60	0.4	46.5	0.0465		Expected number of moles of H2	0.00138
120	0.4	43	0.043		Expected number of moles of Cu	0.00138
180	0.4	39.5	0.0395		Volume of gas collected	0.0377
240	0.4	36	0.036		Pressure of hydrostatic (mm Hg)	1.61765
300	0.4	32.6	0.0326		Pressure of H20 (mm Hg)	19.8
360	0.41	29	0.029		Pressure of H2 (mm Hg)	690.382
420	0.41	25.6	0.0256		Pressure of H2 (atm)	0.9084
480	0.41	22.4	0.0224		Number of moles of hydrogen	0.00141
540	0.41	18.8	0.0188		Moles of Cu oxidized	0.00154
600	0.41	15.5	0.0155		% yield of H2	102.238
660	0.41	12.2	0.0122		% yield of Cu	111.581
Average Current:	0.404166667				Difference between buret + beaker	22
					Pressure of Air	711.8
Initial Volume:	49.9		0.0499		R Value	0.0821
Final Volume:	12.2		0.0122		Temperature	295.2
Change in Volume	37.7		0.0377			
Initial Mass (grams)	4.221					
Final Mass (grams)	4.123					
Molar Mass of Copper	63.546					

Figure 7- Trial 6 of the experiment performed by Melissa Marx with electrode air drying on Kim Wipe

This trial was performed by myself and was done differently than the previous trials. With clear indication that there are issues with the amount of copper being lost when dried, a new method was used to attempt to lose less. This method was using a Kim Wipe for the copper electrode to dry on and then transfer it into the balance to record the mass. When doing so, mass was unfortunately still lost off of the electrode as it stuck on the Kim Wipe. This helps to explain the 111% yield that is shown above for copper. The following trials will explore different options to avoid this loss.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	265.091
0	0.4	49.8	0.0498		Number of moles of electrons	0.00275
60	0.41	46.5	0.0465		Expected number of moles of H2	0.00137
120	0.42	42.5	0.0425		Expected number of moles of Cu	0.00137
180	0.43	39	0.039		Volume of gas collected	0.0364
240	0.43	35.4	0.0354		Pressure of hydrostatic (mm Hg)	2.42647
300	0.44	32	0.032		Pressure of H20 (mm Hg)	19.8
360	0.45	28.1	0.0281		Pressure of H2 (mm Hg)	689.574
420	0.45	24.9	0.0249		Pressure of H2 (atm)	0.90733
480	0.47	21.6	0.0216		Number of moles of hydrogen	0.00136
540	0.46	17.4	0.0174		Moles of Cu oxidized	0.00121
600	0.5	13.4	0.0134		% yield of H2	99.2136
					% yield of Cu	88.2196
Average Current:	0.441818182				Difference between buret + beaker	33
					Pressure of Air	711.8
Initial Volume:	49.8		0.0498		R Value	0.0821
Final Volume:	13.4		0.0134		Temperature	295.2
Change in Volume	36.4		0.0364			
Initial Mass (grams)	4.022					
Final Mass (grams)	3.945					
Molar Mass of Copper	63.546					

Figure 8- Trial 7 completed by Melissa Marx allowing the copper electrode to completely air dry

This was the seventh trial of the experiment that was completed by myself. The main goal of this experiment was to ensure that the copper electrode was completely dried before the mass was acquired. Given the data above, it is clear that the copper mass that was lost was only an 88% yield. This is the first trial in which copper is well below 100%. I would consider this a valid trial as both the hydrogen gas and the copper was under 100%. In completely air drying the electrode, this allows for a more accurate percent yield as none was lost via paper towel or Kim Wipe.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	267.106
0	0.28	49.5	0.0495		Number of moles of electrons	0.00277
60	0.29	47.6	0.0476		Expected number of moles of H2	0.00138
120	0.29	45.3	0.0453		Expected number of moles of Cu	0.00138
180	0.28	42.4	0.0424		Volume of gas collected	0.0378
240	0.28	39.8	0.0398		Pressure of hydrostatic (mm Hg)	1.02941
300	0.28	37.8	0.0378		Pressure of H20 (mm Hg)	21.1
360	0.28	34.9	0.0349		Pressure of H2 (mm Hg)	694.471
420	0.28	32.6	0.0326		Pressure of H2 (atm)	0.91378
480	0.28	30.2	0.0302		Number of moles of hydrogen	0.00142
540	0.28	27.8	0.0278		Moles of Cu oxidized	0.00151
600	0.28	25.8	0.0258		% yield of H2	102.7
660	0.28	23.2	0.0232		% yield of Cu	109.158
720	0.27	21.9	0.0219			
780	0.27	18.6	0.0186			
840	0.27	16.8	0.0168			
900	0.27	14.2	0.0142			
960	0.27	12	0.012			
Average Current:	0.278235294				Difference between buret + beaker	14
					Pressure of Air	716.6
Initial Volume:	49.8		0.0498		R Value	0.0821
Final Volume:	12		0.012		Temperature	296
Change in Volume	37.8		0.0378			
Initial Mass (grams)	3.996					
Final Mass (grams)	3.9					
Molar Mass of Copper	63.546					

Figure 9- Trial completed by Ryan Elliot, Eli Cordova, Wyatt Niedziejko, Hayden Yeneic, and Melissa Marx

This trial was done with four general chemistry students in order to assist in proving that this lab is suitable for entry level chemistry students. After gaining some recommendations on the wording of the lab, the students began to perform it with little difficulties. The biggest issue that they claimed to have was getting the solution up into the buret with the rubber suction bulb. Looking at their results from Figure 9, it is important to note that when they completed this trial, they placed the electrode onto a paper towel to let it dry. In doing so, they lost some of the copper mass on the paper towel and therefore this explains their relatively high percent yield. With this in mind, we made some changes for the final trial that they completed which is seen in Figure 10.

Time (seconds)	Current (Amps)	Volume (mL)	Volume (L)		Total Charge (coulombs)	285.12
0	0.46	49.4	0.0494		Number of moles of electrons	0.00295
60	0.47	45.5	0.0455		Expected number of moles of H2	0.00148
120	0.48	41.7	0.0417		Expected number of moles of Cu	0.00148
180	0.51	37.7	0.0377		Volume of gas collected	0.0378
240	0.51	33.5	0.0335		Pressure of hydrostatic (mm Hg)	0.95588
300	0.54	29.9	0.0299		Pressure of H20 (mm Hg)	21.1
360	0.56	25	0.025		Pressure of H2 (mm Hg)	694.544
420	0.57	20.9	0.0209		Pressure of H2 (atm)	0.91387
480	0.59	16.4	0.0164		Number of moles of hydrogen	0.00142
540	0.59	12	0.012		Moles of Cu oxidized	0.00134
					% yield of H2	96.2217
					% yield of Cu	90.5441
Average Current:	0.528				Difference between buret + beaker	13
					Pressure of Air	716.6
Initial Volume:	49.8		0.0498		R Value	0.0821
Final Volume:	12		0.012		Temperature	296
Change in Volume	37.8		0.0378			
Initial Mass (grams)	3.9					
Final Mass (grams)	3.815					
Molar Mass of Copper	63.546					

Figure 10- Trial 9 completed by Ryan Elliot, Eli Cordova, Wyatt Niedziejko, Hayden Yeneic, and Melissa Marx

This was the second and final trial completed by the first year chemistry students. This trial is by far the best one that has been completed for this project. Both the percent yields of hydrogen gas and the copper electrode are under 100% which shows that no mass of the electrode was lost from drying. The method used in this process was to air dry the electrode for 10 minutes before going to retrieve its mass. Moving forward, any and all trials should be done so to see if the results are reproducible.

Discussion:

This lab was performed by myself, a senior chemistry student (Kaylin Brant), and four freshmen General Chemistry students. The data above shows that there are variable percent yields which indicates that there is some error within the lab itself. One of the main sources of error would be from the copper electrode. For the first trial that was done, an old copper electrode was used that was previously used in another electrolysis experiment. In being previously oxidized, the results were not what I wanted them to be when it came to having over 100% yield. Even after sanding the old electrode down, it still produced subpar results with copper percent yield over 100 and hydrogen gas below 90 percent. For these reasons, it was decided that if this lab is to be implemented in the curriculum there needs to be the use of copper foil.

Another aspect of this lab that is considered to be a difficult would be collecting all of the hydrogen gas. For this lab, in order to be cost effective, it is easiest to collect the gas in an inverted buret. The issue with this is that since it is a buret, it is typically used to dispense liquid, not collect gas. Even when the buret is closed, there is still the ability of gas to escape out of the end. In order to provide the best way to collect hydrogen gas, the department could look into buying different glass collection tubes. If the top of the tube is completely sealed, the gas can only push the meniscus level down and not escape out the top. On Flinn Scientific, gas collection tubes can be purchased for $20.80 that are closed tubes with the buret readings labelled from top to bottom (Picture 1).

Picture 1- Glass Measuring Tube, Borosilicate Glass, 50 mL. Note the closed, rounded top.

https://www.flinnsci.com/gas-measuring-tube-borosilicate-glass-50-ml/gp5067/

If these are bought, this also eliminates the issue of reading a buret upside down. If new gas collection tubes are bought, this will help to ensure that the hydrogen gas that is being produced isn't escaping from the tube.

Most of the issues in this lab come from the percent yield of the copper electrode regardless of whether it was the foil or the previously used electrode. While the copper foil appeared to yield better results, there were still issues since over 100% of copper was being lost in this experiment. One of the main reasons for this could be due to the method of drying it. In trials 1-5, the electrode was dried via Kim Wipe. In doing this, some of the copper was taken off of the electrode and created a greater loss than what was done in the redox reaction itself. For

this reason, drying the electrode was further studied with the general chemistry students. These groups were told to let the copper electrode to air dry and measure its mass 3 times over the course of 10 minutes to ensure that an accurate mass was obtained.

Conclusion:

With all of the given trials, the electrolysis lab that has been in question has the means to fit in the curriculum at Alfred University. After performing several different runs with different students, this electrolysis lab has great potential in being a lab for undergraduate chemistry students. This is confirmed by the success of trials done by the General Chemistry students. Even with some sources of error, this lab provides the necessary learning opportunities for students in hands on chemistry, observations, and calculations. The addition of this lab will greatly benefit the future students as well as strengthening the department's variety of general chemistry labs.

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