The Development of an Electrochemical Experimental Procedure for use in an Analytical Chemical Course.

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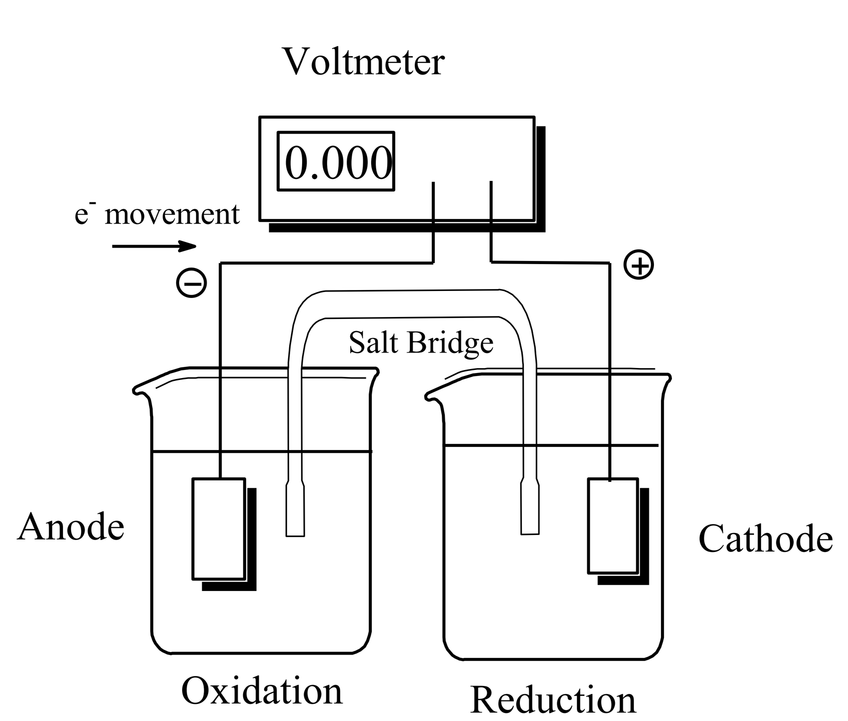
Introduction

Students often find themselves frustrated by experiments in science courses when the experiments fail. Sometimes the directions are vague or confusing, sometimes the design of the experiment is flawed. Sometimes there is insufficient guidance because the instructor hasn't written the lab themselves or tried the experiment exhaustively to ensure that they can guide the student through common pitfalls or challenges. A benefit of having a student researcher work on this is to get a student perspective of the procedure

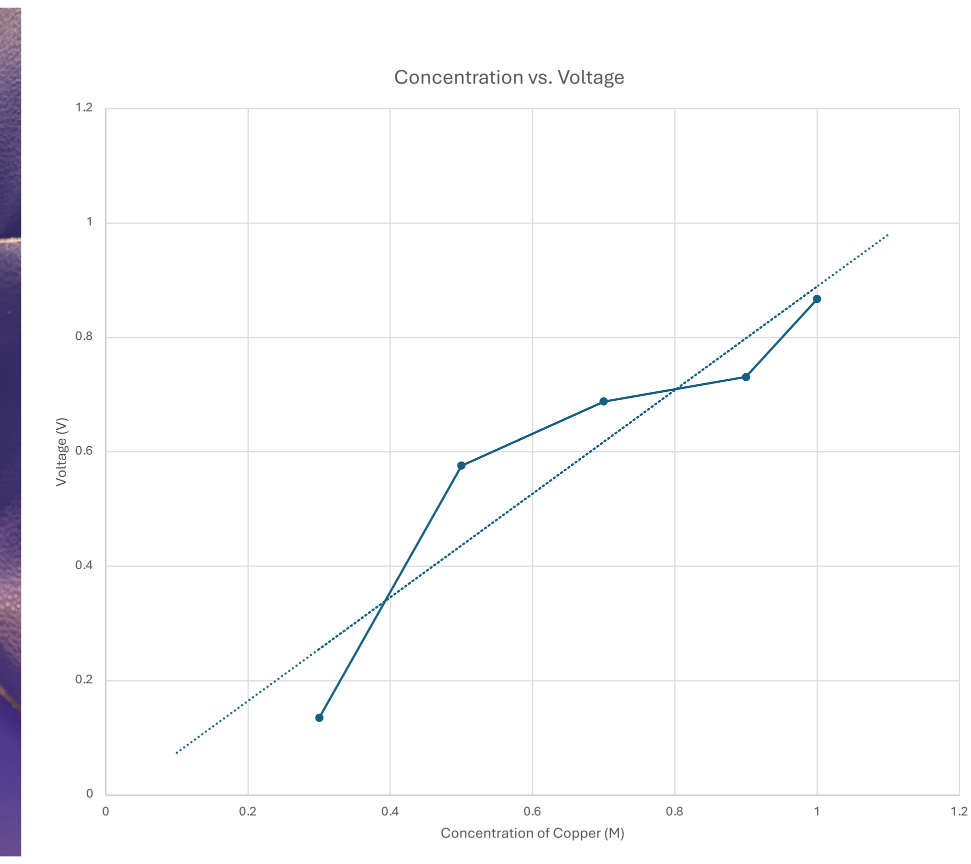
Methods

The kind of electrochemistry experiments that are described in the analytical chemistry course are based around the galvanic cell. The galvanic cell, pictured, has two beakers that contain chemical solutions that are capable of oxidation and reduction processes. As the solutions react, the ions must gain or lose electrons to change their oxidation state. Each of the containers has some of the ion in solution, and an electrode of an appropriate material to function as the electron transfer medium. Since the solutions are in separate containers the apparatus uses a voltmeter to measure voltage change, and to transfer the electrons from one solution to the other. At the same time, the anode has atoms that are being oxidized which will dissolve into an ionic state while the cathode has atoms which are deposited onto the metal surface as they are being reduced.



Labeled drawing of an electrochemical cell

Electrochemical cell with
Cu(s) | Cu^{2+} (.125 M) || Zn^{2+} (.1M) | Zn(s)



Graph of Data using
Cu(s) | Cu^{2+} (0.3-1M) || Zn^{2+} (1M) | Zn(s)

Results

Reaction (0.1M)	Voltage (Volts)
Cu(s) | Cu^{2+} (.1 M) || Zn^{2+} (.1 M) | Zn(s)	0.790 V
Cu(s) | Cu^{2+} (.1 M) || Ni^{2+} (.1 M) | Ni(s)	0.148 V
Fe(s) | Fe^{2+} (.1 M) || Ni^{2+} (.1 M) | Ni(s)	0.331 V
Fe(s) | Fe^{2+} (.1 M) || Cr^{3+} (.1 M) | Cr(s)	0.267 V

Reaction (1.0M)	Voltage (Volts)
Fe(s) | Fe^{2+} (1 M) || Cr^{3+} (1 M) | Cr(s)	0.549 V
Zn(s) | Zn^{2+} (1 M) || Cr^{3+} (1 M) | Cr(s)	0.749 V
Ni(s) | Ni^{2+} (1 M) || Cr^{3+} (1 M) | Cr(s)	0.839 V
Cr(s) | Cr^{3+} (1 M) || Cu^{2+} (1 M) | Cu(s)	0.215 V
Ni(s) | Ni^{2+} (1 M) || Cu^{2+} (1 M) | Cu(s)	0.182 V
Zn(s) | Zn^{2+} (1 M) || Cu^{2+} (1 M) | Cu(s)	0.867 V
Fe(s) | Fe^{2+} (1 M) || Cu^{2+} (1 M) | Cu(s)	0.626 V
Fe(s) | Fe^{2+} (1 M) || Ni^{2+} (1 M) | Ni(s)	0.260 V
Fe(s) | Fe^{2+} (1 M) || Zn^{2+} (1 M) | Zn(s)	0.585 V
Zn(s) | Zn^{2+} (1 M) || Ni^{2+} (1 M) | Ni(s)	0.802 V

Our next stage of the experiment was to make up solutions of varying concentration so we could test the voltages to see if the changes in voltage are consistent and if the changes graph in a linear fashion. If this is the case, then we should be able to use the electrochemical cell apparatus to measure the voltage of an unknown concentration of the ion and use the equation below to calculate the concentration of the solution.

$$E_{cell} = E^{\circ}_{cell} - \left(\frac{0.0592\ V}{n}\right) \log\left(\frac{[Ox]}{[Red]}\right)$$

Conclusion

A useful experiment for an analytical chemistry lab course is one that would allow for the identification of an unknown concentration of an ion. Experiments of this type are commonly achieved using very expensive pieces of equipment or complicated scientific instruments. UV-vis experiments that can measure absorbance of light in the UV range can be used for this process, but these instruments can cost thousands of dollars. Most of the equipment in an electrochemical cell is very inexpensive. These items include common laboratory glassware, like beakers. The U-shaped tube is only a few dollars to purchase, and a high-quality voltmeter can be purchased for less than $50. The only remaining costs are for the appropriate electrodes, which are commonly available and the chemical salts, many of which are already used in the existing course.

This work is continuing but should be completed by the end of the current semester. This should allow us to try the experiment as part of the current offering of analytical chemistry. Assuming that the experiment is successful, Dr. Delgado will be able to add this experiment to all future offerings of this class.

Electrochemical constants and equations are from:
Harris, D. C.; Lucy C. A. *Quantitative Chemical Analysis*, Macmillian Learning, 10th ed., 2020