

The Analysis of Electrochemical Properties of Metal Protoporphyrins for the Reduction of Carbon Dioxide Via Engineered Metalloproteins, or, How to Not Flood Beeghly

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Abstract

This Capstone examines a semester spent as a research assistant with American University's Chemistry Department both technically and creatively. The first portion of the project is an analysis and report on the reductive properties of heme and other metal protoporphyrins. Heme b, the molecule found inside the protein hemoglobin that gives blood its distinctive red coloring, has been proven to be effective at transporting electrons, and this capstone seeks to identify how this molecule transforms carbon dioxide into less harmful compounds, such as formic acid and carbon monoxide, when in solution. This research explores similar molecules- cobalt, manganese, copper, and nickel protoporphyrins- and their ability to reduce carbon dioxide inside a modified protein. These findings were gathered via Ultraviolet-Visible spectroscopy and cyclic voltammetry. The second portion of this Capstone, a collection of essays titled "She Blinded Me With Science: Boiling Acetic Acid Outside of the Fume Hood and Other Beeghly Mishaps and Musings," contemplates the less concrete findings that come from working in the lab. From lab floods to grad school rejections to indecipherable Halloween costumes, this section of the Capstone hopes to prove that there's room for humor in analytic chemistry.

To Cake Day

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The Analysis of Electrochemical Properties of Metal Protoporphyrins for the Reduction of Carbon Dioxide Via Engineered Metalloproteins

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Abstract

The protein hemoglobin contains iron heme, a metal protoporphyrin that is used in electron-transfer processes, and is responsible for transporting oxygen through the vascular system when in hemoglobin. Using the known reductive properties of similar molecules, such as iron tetraphenylporphyrin, further electrochemical tests were performed in order to identify similar properties in iron-manganese-, cobalt-, copper-, and nickel-protoporphyrins. UV-Vis spectroscopy was run on the metal protoporphyrins, and the absorbance levels of nickel solutions of varying concentrations was observed in particular. Cyclic voltammetry was also performed on solutions of ferrocene aldehyde, t-butylhexafluorophosphate, and iron protoporphyrin IX. Results of the study were not conclusive, and further experiments are required in order to identify the ability of metal protoporphyrin to reduce carbon dioxide both in solution and inside a modified protein. Recommended changes to future experiments include additional UV-Vis spectroscopy studies in order to find the definitive molar absorptivity of metal protoporphyrin solutions, as well as the addition of ascorbic acid to solutions in order to maintain an absorbance level below 1.0. The cyclic voltammetry experiments are also recommended for additional trials, and each metal protoporphyrin should be examined.

Introduction

Hemoglobin and Metal Porphyrins

The metalloprotein hemoglobin performs essential functions for the human body, most notably the transportation of oxygen throughout the bloodstream to vital organs. Hemoglobin (Figure 1) is comprised of four globular subunit proteins, held together by hydrogen bonds that create a globin fold arrangement. Inside this folding arrangement, giving the distinctive spirals of the protein lies the heme, an iron-containing porphyrin group (Kosmachevskaya and Topunov, 2008).

The heme group in hemoglobin is often referred to as iron protoporphyrin IX (Figure 2). Protoporphyrin is the end-product of the metabolism of porphyrins, aromatic, cyclic molecules comprised of four pyrrole subunits connected by methane bridges at their α carbons. Protoporphyrins can bind with other metals to form compounds, but the most common form is heme b. A molecule very similar to heme b, iron tetraphenylporphyrin, has shown reductive properties and effective electron transfer, showing its potential to reduce carbon dioxide when placed in a photosynthetic center. In their paper "Proton-coupled Electron Transfer Reactions at a Heme-Propionate in an Iron-Protoporphyrin-IX Model Compound," Jeffrey J. Warren and James M. Mayer performed an experiment in which $(\text{PPIX}_{\text{MME}})\text{Fe}^{\text{III}}(\text{MeIm})_2$ propionate is reduced in an acetonitrile solution via proton-coupled electron transfer and concerted proton-

electron transfer, with protons being transferred at the heme propionate site and electrons being transferred at the iron center itself (Mayer and Warren, 2011). David L. Compton and Joseph A. Laszlo examine the same phenomenon in their paper “Direct electrochemical reduction of hemin in imidazolium-based ionic liquids,” showing electrochemical activity of heme when observed via cyclic voltammetry (Compton and Laszlo, 2002).

Similarly, in “Surface Raman Scattering and Electrochemistry of Iron Protoporphyrin IX at a Polycrystalline Silver Electrode,” J.J. McMahon, S. Baer, and C.A. Melendres show iron protoporphyrin IX to be able to effectively reduce O_2 (Baer, McMahon, and Melendres, 1986). This study, however, seeks to examine the reactivity of hemin and other metal protoporphyrins with carbon dioxide. Numerous studies have shown iron tetraphenylporphyrin and other similar molecules to be effective catalysts for carbon dioxide reduction in the presence of triethylamine, following a step-wise mechanism for electron transfer without being consumed themselves (Fujita, 1998) (Behar, Grodkowski, Hambright, and Neta, 1997). Metal porphyrins often decrease the overpotential for the reduction reaction for carbon dioxide to form formic acid and carbon monoxide from -2.0 V to -1.0 V, showing an increased efficiency in the system and an improvement in the quality of the catalyst. Particular successes have been found with cobalt protoporphyrin, having an electroreduction efficiency rate of 64% (Bandhopadhyay, Jha, Phougat, and Vasudevan, 2002) (Leung and Nielsen, 2010).

This research aims to explore which metallic porphyrins have electrochemical properties that best reduce carbon dioxide. While iron and cobalt tetraphenylporphyrin has been proven to reduce carbon dioxide, this study seeks to compare the reactivity of iron heme and carbon dioxide with those of other

metallic protoporphyrins both inside and outside a modified protein. Manganese, cobalt, copper, and nickel were chosen as the metals to test due to their similar electronegativity and conductivity to iron. The second part of the experiment, which will not be covered in this paper, will be to modify the structure of the protein in order to best accommodate the metal porphyrin and increase the rate of reactivity by adding photosensitizers.

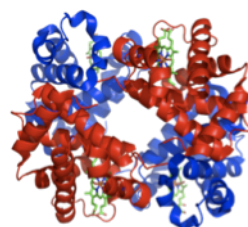


Figure 1. Hemoglobin

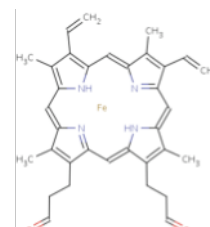


Figure 2. Iron Protoporphyrin IX

Electrochemical Techniques

Electrochemical properties of a solution can be determined through several means, one of which is Ultraviolet-Visible spectroscopy. UV-Vis spectroscopy measures either the level of absorbance that occurs when light is passed through a solution. In order to do this, two light sources are utilized. The first is a light in the UV spectrum from 200-400 nm, powered by a deuterium or hydrogen light source. The second is in the visible spectrum; tungsten filaments are often used for this light source. The lights are sent through a prism, which separates the spectrum into their constituent colors, and the light is then bounced off a series of mirrors into both a reference cuvette and sample cuvette. The reference cuvette provides the absorbance data for just the solvent, while the sample cuvette provides the data for the solution being tested. The absorbance is found through the transmittance ratios of the two cuvettes. (Mohan, 2002)

When the light interacts with a molecule that matches the difference between the electronic energy levels in that molecule, the molecule absorbs the energy from that wavelength, using the energy to move electrons from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO), creating an excited state. This absorbance at a specific wavelength is what is recorded on a UV-Vis graph, and can display electrochemical properties about the molecule. The amount of light absorbed, and at which wavelength, is determined by the placement of pi bonds, as well as the amount of delocalization. As delocalization increases, so does the wavelength where maximum absorbance occurs (Stanfield, 2001). That said, an ideal solvent for UV-Vis spectrometry is one that does not have conjugated pi bonds that could interfere with the reading (Mohan, 2002). While water and n-hexane are common solvents, dimethylformamide (Figure 3) will be used in this experiment.

Dimethylformamide is a polar substance with donor electrons at both the oxygen and nitrogen atoms, has been proven to be an effective solvent for iron protoporphyrin IX in UV-Vis spectrometry (Makarska-Bialokoz, Pratiel, and Radzki, 2007).

Dimethylformamide is also a commonly used solvent in UV-Vis due to its low cut-off wavelength at only 270 nm, before the visible spectrum. An effective UV-Vis procedure calls for 0.1-100 mg of solute to be used.

Cyclic voltammetry can also be used to determine electrochemical properties, most notably, the amount of energy required to run a reaction. Cyclic voltammetry is a means of potentiodynamic electrochemical measurement, and is used to determine the reductive and oxidative potentials of a solution, and to determine whether a reaction is reversible (Heineman and Kissinger, 1983). A certain potential is applied to the system up to a certain voltage for the cathodic reading; at that point, the potential is reversed, and the anodic reading is recorded (Fry, 1989). Three

main electrodes are used in cyclic voltammetry: a reference electrode, a working electrode, and a counter-electrode. The reference electrode, usually comprised of a solution of silver nitrate, works to measure the potential with the working electrode, an insulator usually comprised of glassy carbon or platinum. The working electrode and the counter-electrode, which maintains current, measure the current of the system. Successful previous experiments using cyclic voltammetry to measure the electrochemical properties of iron protoporphyrin IX have used platinum as a counter-electrode (Mayer and Warren, 2011).

During a cyclic voltammetry experiment, the current is plotted against the applied voltage. A successful run will give the distinctive graph seen in Figure 4. A cyclic voltammogram can reveal many electrochemical properties, the first being reversibility. A reaction is said to be reversible if:

$$| E_{pc/2} - E_{pa/2} | = 57 \text{ mV}/n$$

where n is the scan rate. E_{pc} denotes the cathodic potential peak, and E_{pa} denotes the anodic potential peak. The averages of these values give the half-point potentials.

Reversibility can also be determined by examining the anodic and cathodic peak currents, where if $i_{pa}/i_{pc} = 1$, the system is said to be reversible (Fry, 1989). A non-reversible cyclic voltammogram points to an electric energy that has altered the catalyst, and thus rendered the reaction ineffective.

The purpose of this experiment is to explore the electrochemical properties of various protoporphyrins through Ultraviolet-Visible spectrometry and cyclic voltammetry. These properties will be analyzed to find a suitable substitute for the iron heme in hemoglobin in order to best catalyze carbon dioxide.

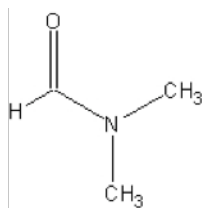


Figure 3. Dimethylformamide

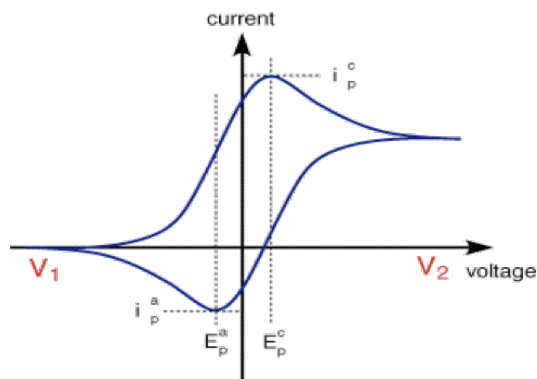


Figure 4. Typical cyclic voltammogram plotting a reversible reaction

Materials and Methods

Part I. UV Spectrometry of Metal Protoporphyrins

A. Original

2 mg of Fe protoporphyrin IX were added to a cuvette. 1000 μ L of dimethylformamide were then added to the cuvette. A dilution of the solution was made with the 10 μ L of the previously mixed solution and 1 μ L of dimethylformamide. The cuvette was then run through a UV spectrometer reading, with the scanner set the 200-800 nm at 1.0 nm intervals. The results were recorded. The same procedure was repeated for Mn protoporphyrin IX, Co protoporphyrin IX, Ni protoporphyrin IX, and Cu protoporphyrin IX.

B. Dilutions

2 mg of Fe protoporphyrin IX were added to a cuvette. 1000 μ L of dimethylformamide were then added to the cuvette. A dilution of the solution was made with the 2.5 μ L of the previously mixed solution and 1 μ L of dimethylformamide. The cuvette was then run through a UV spectrometer reading, with the scanner set the 200-800 nm at 1.0 nm intervals. The results were recorded. The same procedure was repeated for Mn protoporphyrin IX, Co protoporphyrin IX, Ni protoporphyrin IX, and Cu protoporphyrin IX.

C. Dilutions of Nickel

2.5 mg of Ni protoporphyrin IX were mixed with 10 μ L of dimethylformamide to make a 3.8×10^{-5} M solution. From that stock solution, solutions of 4.2×10^{-6} M, 4.2×10^{-7} M, 2.1×10^{-7} M, 1.8×10^{-7} M, and 3.9×10^{-8} M were made and added to cuvettes. The cuvettes were then run through a UV spectrometer reading, with the scanner set the 200-800 nm at 1.0 nm intervals. The results were recorded. The procedure was then repeated with solutions of Ni protoporphyrin IX, dimethylformamide, and ascorbic acid.

D. Dilutions of Nickel in a Light Intensity UV Spectrometer

The solutions from Part 1.C were prepared, and then into a light intensity UV spectrometer. The integration time was set to 8 milliseconds, and the scan rate was set to 25. The results were recorded.

Part II. Cyclic Voltammetry

A. Purification of Fe Protoporphyrin

In order to prepare the hemin sample for UV-Vis spectrometry or cyclic voltammetry, the hemin must be purified in order to remove any impurities that may interfere with the electrochemical measurements or scatter light,

giving inaccurate results. 1.11 g of iron heme was shaken in 25 mL of pyridine until dissolved. 8 mL of CHCl_3 were added to the solution. The beaker was shaken on a stirring plate for five minutes. The solution was then filtered through a Buchner funnel and washed with 2 mL of CHCl_3 . A saturated NaCl solution was made by boiling 26.2 g of NaCl in 100 mL of water. 40 mL of acetic acid were heated to boiling, and 1 mL of NaCl and HCl were added. The CHCl_3 solution was poured into the acetic acid solution while stirring, and set aside for 12 hours. The crystals were filtered off, and washed with a solution of acetic acid, H_2O , EtOH, and Et_2O , and dried in air. The crystals were then massed and recorded.

B. Recrystallization of Tetra-butyl-hexafluorophosphate

Cyclic voltammetry requires the conduction of current, necessitating an electrolyte. In this section, tetra-butyl-hexafluorophosphate was produced in order to act as an electrolyte in the system. 2 millimoles of NH_4PF_6 were added to a beaker and mixed with just enough water to dissolve the solute. 2 millimoles of tetra-n-butylammonium chloride were added to another beaker and mixed with just enough water to dissolve the solute. The solutions were then mixed together, and the crystals were collected from a Buchner funnel. The crystals were dissolved in a ethanol, and the ethanol solution was heated until boiling. The solution was then placed in an ice bath until crystals reformed. The crystals were then run through a Buchner funnel once more. The crystals were then massed, and the results recorded.

C. Production of Ni Protoporphyrin

10 mL of dimethylformamide was added to a round-bottom flask and heated until reflux at 85^a Celsius. 0.5 g of protoporphyrin was added to the dimethylformamide until dissolved. 0.14 g nickelous sulfate, an equally

molar amount, was added to the solution. When solute had completely dissolved, the solution was checked with a UV lamp for red fluorescence. The solution was then chilled in ice water for 15 minutes. 100 mL of chilled water was then added to the solution. The solution was run through a Buchner funnel and rinsed with water. The precipitate was massed, and the results recorded.

D. Cyclic voltammetry of Fe Protoporphyrin IX

0.013 g of ferrocene aldehyde and 0.4 g of t-butyl-ammonium hexafluorophosphate were added to 10 mL of acetonitrile. The solution was then stirred on a stirring plate. Nitrogen was bubbled through the system continuously for 30 minutes. The solution was run through a cyclic voltammetry test with a reference electrode filled a 0.1 M solution of silver nitrate in acetonitrile. The test was run in 2 segments, with a potential range from 1 V to -1 V, and a 100 mV/s sweep rate. The results were recorded, and the same procedure was repeated for a solution of acetonitrile, tetra-butyl-ammonium hexafluorophosphate, and Fe protoporphyrin IX of equal molarity.

Results

Part 1. UV Spectrometry of Metal Protoporphyrins

The first round of UV-Vis spectrometry examined the absorbance of various metal protoporphyrins, as shown in Chart 1. All five of the metal protoporphyrins followed a similar shape, peaking between 391 and 409 nm and quickly tapering off. Fe peaked at 391 nm, Co peaked at 409 nm, and Cu, Mn, and Ni peaked at 406 nm. Fe, Co, Cu, and Ni protoporphyrin all had a maximum absorbance between 3.540 and 3.725, with Fe absorbing at 3.672, Co absorbing at 3.723, Cu absorbing at 3.725, and Ni absorbing at 3.54. Mn had a maximum absorbance of 1.475, the lowest of the metal protoporphyrins. Fe had

the broadest spectrum, as well as fewer variations in absorbance after 450 nm. All of the metal protoporphyrins experienced a severe drop, and then continual oscillation between an absorbance of 0 and 0.6 from 0-261 nm.

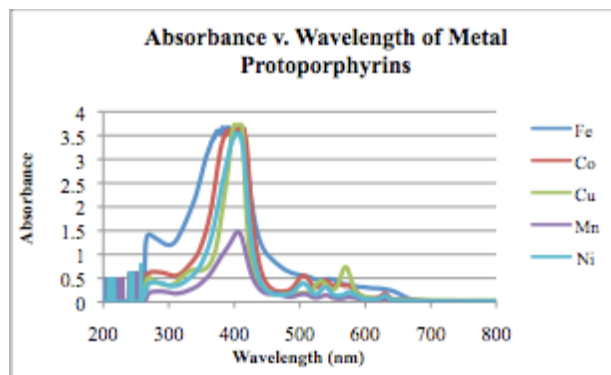


Chart 1. Absorbance v. Wavelength of Metal Protoporphyrins when run through a UV spectrometer (Materials and Methods 1.A)

The second experiment examined a 25% dilution of the metal protoporphyrin solutions. Again, the solutions, as shown in Chart 2, reached sharp, parabolic apexes at similar wavelengths, peaking between 394 and 406 nm. Fe had a maximum wavelength of 394 nm, Co had a maximum wavelength of 402 nm, Cu had a maximum wavelength of 404 nm, Mn had a maximum wavelength of 406 nm, and had a maximum wavelength of 400 nm. All five metal protoporphyrins had distinct maximum absorbance levels. Fe had a maximum absorbance of 1.331, Co had a maximum absorbance of 0.953, Cu had a maximum absorbance of 1.731, Mn had a maximum absorbance of 0.475, and Ni had a maximum absorbance of 3.425. All of the metal protoporphyrins experienced a severe drop at 249 nm, oscillating between an absorbance level of 0.202 and 0.802. Only the Ni protoporphyrin experienced both increasing and decreasing absorbance levels after 500 nm, finally reaching a steady decrease at 658 nm, as shown in Chart 3.

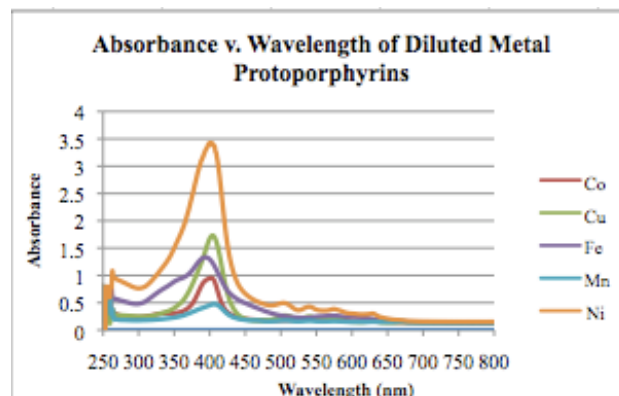


Chart 2. Absorbance v. Wavelength of Metal Protoporphyrins with a 25% Dilution (Materials and Methods 1.B)

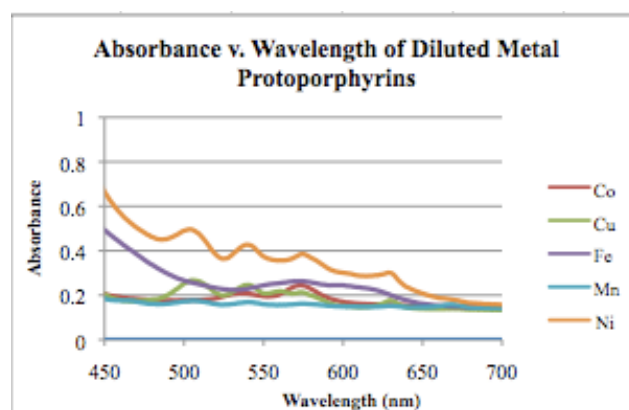


Chart 3. Absorbance v. Wavelength of Metal Protoporphyrins with a 25% Dilution (Materials and Methods 1.B) from 450-700 nm

For the diluted metal protoporphyrins, Ni had the highest molar absorptivity at the maximum wavelength, followed by Cu, then Fe, then Co, and finally, Mn, as shown in Chart 4. Ni had a molar absorptivity of $9.86 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, Cu had a molar absorptivity of $5.02 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, Fe had a molar absorptivity of $3.82 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, Co had a molar absorptivity of $2.75 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, and Mn had a molar absorptivity of $1.36 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$.

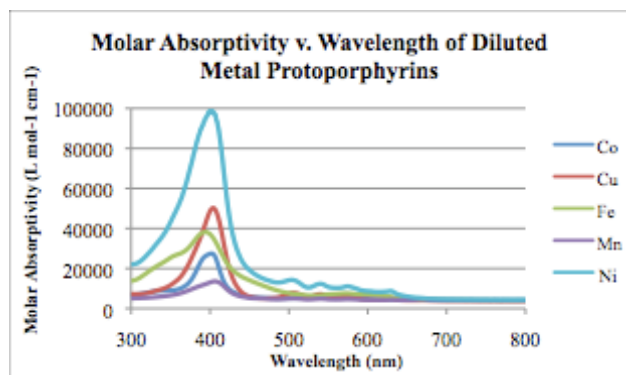


Chart 4. Molar Absorptivity v. Wavelength of Diluted Metal Protoporphyrins

The third part of the UV-Vis analysis of the electrochemical properties of metal protoporphyrins examined Ni protoporphyrin solutions with varying concentrations, as shown in Chart 5. The solutions had similar maximum wavelengths: the 4.2×10^{-6} M solution had a broad peak between 402 and 421 nm, the 4.2×10^{-7} M solution had a peak at 405 nm, and the 2.1×10^{-7} M, 1.8×10^{-7} M, and 3.9×10^{-8} M solutions had maximum wavelengths of 406 nm. Each concentration had a distinct maximum absorbance. The maximum absorbance was between 3.785 and 3.787 nm for the 4.2×10^{-6} M solution, 1.181 for the 4.2×10^{-7} M solution, 0.694 for the 2.1×10^{-7} M solution, 0.380 for the 1.8×10^{-7} M solution, and 0.276 for the 3.9×10^{-8} M solution. The solutions all dropped off severely at 260 nm, oscillating between an absorbance level of 0.0 and 0.3. Only the most concentrated solution showed the peaks and valleys in absorbance levels between 500 and 650 nm.

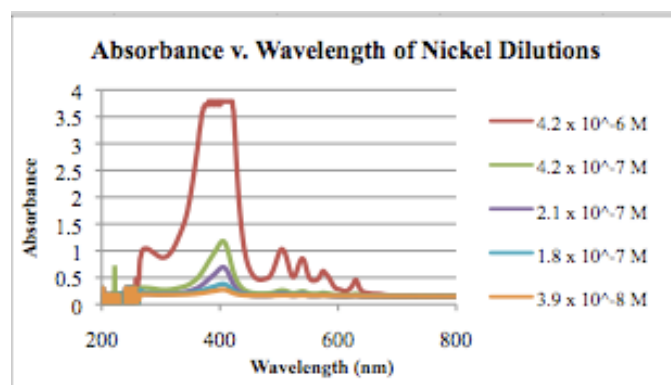


Chart 5. Absorbance v. Wavelength of Nickel Protoporphyrin at Various Concentrations (Materials and Methods 1.C)

From this data, the molar absorptivity of each solution was found using Beer's Law:

$$A = \epsilon bc$$

Where A is absorbance, ϵ is the molar absorptivity ($\text{L mol}^{-1} \text{cm}^{-1}$), b is the path length of the cuvette (in this case, 1 cm), and c is the concentration of the compound. Chart 6 shows the molar absorptivity of the solutions in relation to the wavelength. All solutions had a maximum wavelength between 402 and 409 nm, and showed the same parabolic path as Chart 5. Solutions with a lower concentration had a higher absorptivity, with the exception of the 1.81×10^{-7} M solution. The solvent DMF absorbs light at 270 nm and below, and thus skews data; for this reason, the chart begins at 300 nm.

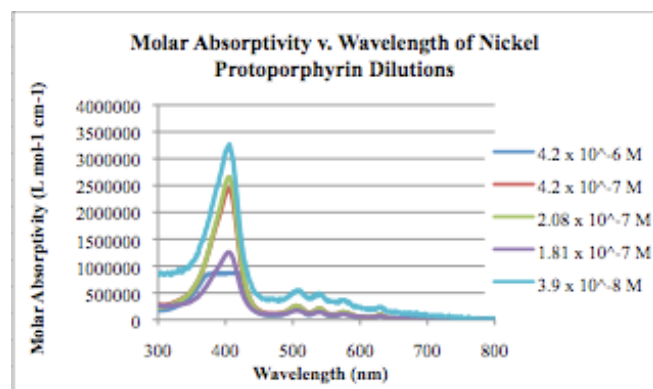


Chart 6. Molar Absorptivity v. Wavelength of Nickel Dilutions (Materials and Methods 1.C)

Nickel protoporphyrin IX dilutions were also performed with the addition of ascorbic acid, a reductive agent. The chart followed the same parabolic path, with each solution reaching a maximum wavelength between 404 and 407 nm, as shown in Chart 7. The 4.2×10^{-6} M solution had a maximum wavelength of 406.05 nm, the 4.2×10^{-7} M had a maximum wavelength of 405.32 nm, and the remaining solutions had a maximum wavelength of 404.6 nm. The 4.2×10^{-6} M solution had the highest absorbance of 0.36, followed by the 4.2×10^{-7} M solution with an absorbance of 0.17, the 1.81×10^{-7} M solution with an absorbance of 0.11, the 2.08×10^{-7} M solution with an absorbance of 0.10, and the 3.86×10^{-8} M solution with 0.07.

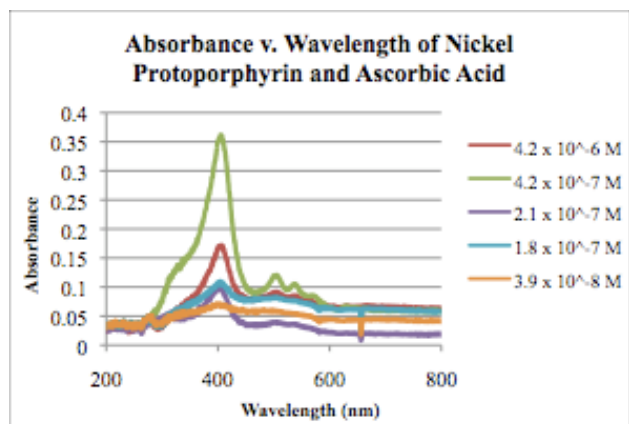


Chart 7. Absorbance v. Wavelength of Nickel Protoporphyrin Dilutions and Ascorbic Acid (Materials and Methods 1.C)

The same nickel dilutions were then run through a UV-Vis spectrometer, which directly measures light intensity, not absorbance. The data was changed to absorbance, and plotted. With the new UV-Vis spectrometer, the data appeared much more irregular, as shown in Chart 8. The 3.86×10^{-8} M solution had an irregular path, beginning to peak at the same maximum wavelength as with UV-Vis 1, and then reaching a plateau at an absorbance level of 0.1. Also irregular was the negative absorbance of the 2.08×10^{-7} M, and the large

valleys and peaks were more pronounced with the 4.2×10^{-6} M solution.

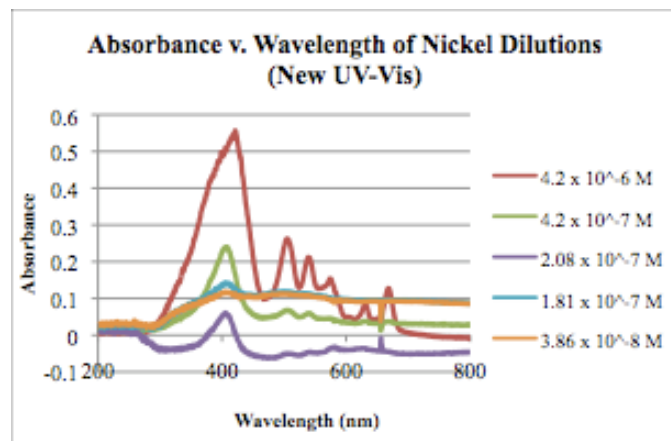


Chart 8. Absorbance v. Wavelength of Nickel Dilutions using new UV-Vis spectrometer (Materials and Methods 1.D)

Part 2. Preparation of Cyclic Voltammetry Analytes and Materials

During the purification of hemin from iron protoporphyrin IX chloride, the hemin formed a fine, deep-violet colored powder when reacted with the CHCl_3 and Acetic acid and filtered through a Buchner funnel. Table 1 shows the actual and theoretical mass of the purified hemin. The actual mass was 0.53 g, and the theoretical mass was 1.1 g,

Table 1. Mass of Purified Hemin

	Mass (grams)
Actual Mass of Purified Hemin	0.53
Theoretical Mass of Purified Hemin	1.1

Percent Yield: 48%

When NH_4PF_6 and tetra-n-butylammonium chloride were combined and dissolved in ethanol, white, malleable crystal-like structures form when the solution was filtered through a Buchner funnel. Table 2 shows the actual and theoretical mass of the precipitate, tetra-butylhexafluorophosphate. The actual mass

was 0.48 g, and the theoretical yield was 0.78 g, giving a percent yield of 62%.

Table 2. Mass of Recrystallized Tetra-butylhexafluorophosphate

	Mass (grams)
Actual Mass of Recrystallized Tetra-butyl-hexafluorophosphate	0.48
Theoretical Mass of Recrystallized Tetra-butyl-hexafluorophosphate	0.78

Percent Yield: 62%

Part 3. Cyclic Voltammetry

Reduction potentials of solutions and reversibility of reactions were measured using cyclic voltammetry. The solution of ferrocene aldehyde and t-butylhexafluorophosphate was run at a scan rate of 25 mV/s had a cathodic peak potential at 0.46 V with a cathodic peak current at 8.47×10^{-5} A, and an anodic peak potential at 0.23 V with an anodic peak current at -5.53×10^{-5} A, as shown in Chart 9. These values did not solve for the reversibility equations, and thus the solution was found to be irreversible.

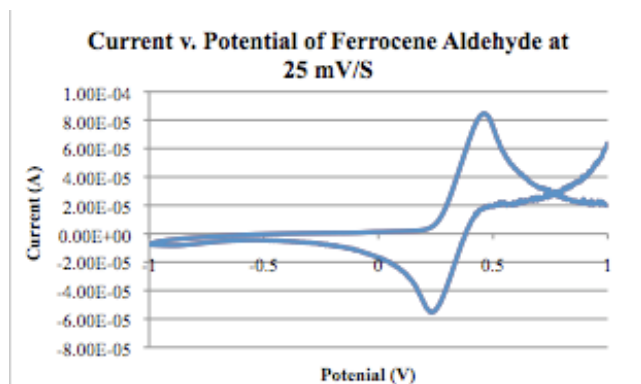


Chart 9. Current v. potential of ferrocene aldehyde and t-butylhexafluorophosphate in acetonitrile solution at scan rate of 25 mV/s (Materials and Methods 2.D)

The solution of ferrocene aldehyde and t-butylhexafluorophosphate was run at a scan rate of 100 mV/s had a cathodic peak potential at 0.51 V with a cathodic peak current at 1.4×10^{-4} A and an anodic peak

potential at 0.19 V with an anodic peak current at -9.43×10^{-5} A, as shown in Chart 10. These values did not solve for the reversibility equations, and thus the solution was found to be irreversible.

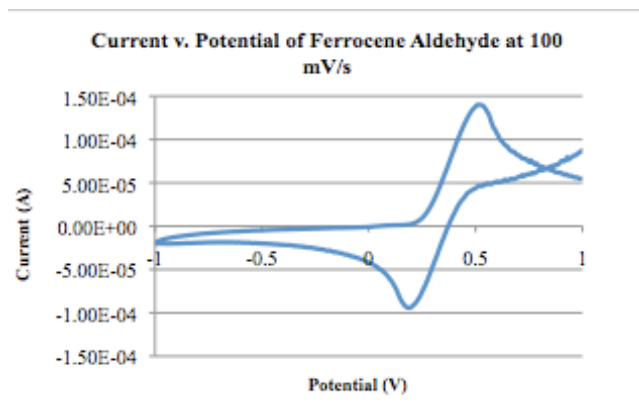


Chart 10. Current v. potential of ferrocene aldehyde and t-butylhexafluorophosphate in acetonitrile solution at scan rate of 100 mV/s (Materials and Methods 2.D)

A solution of ferrocene aldehyde, t-butylhexafluorophosphate, and iron protoporphyrin IX was run at a scan rate of 25 mV/s. The solution reached a cathodic peak potential at 0.51 V with a cathodic peak current at 5.87×10^{-5} A, and an anodic peak potential at 0.24 V with an anodic peak current at -2.3×10^{-5} A, as shown in Chart 11. These values did not solve for the reversibility equations, showing the reaction to be irreversible.

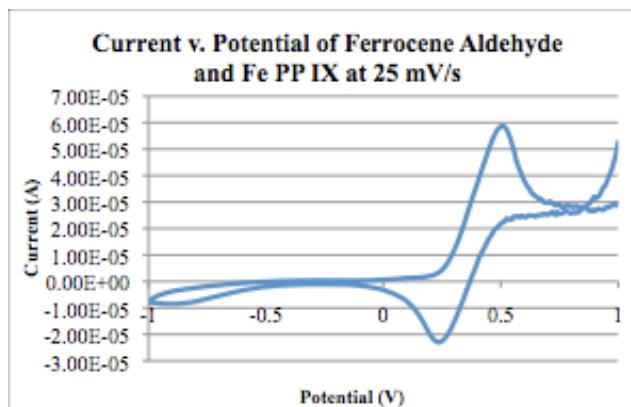


Chart 11 Current v. potential of ferrocene aldehyde, t-butylhexafluorophosphate, and iron protoporphyrin IX in acetonitrile solution at scan rate of 25 mV/s (Materials and Methods 2.D)

A solution of ferrocene aldehyde, t-butylhexafluorophosphate, and iron protoporphyrin IX was run at a scan rate of 100 mV/s. The solution reached a cathodic peak potential at 0.61 V with a cathodic peak current at 1.1×10^{-4} A, and an anodic peak potential of 0.15 V with an anodic peak current of -5.6×10^{-5} A, as shown in Chart 12. These values do not solve for the reversibility equations, showing the reaction to be irreversible.

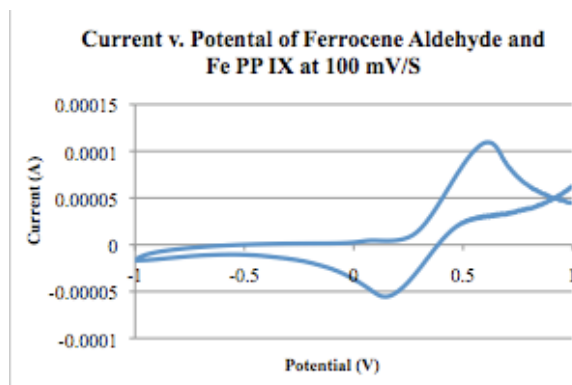


Chart 12. Current v. potential of ferrocene aldehyde, t-butylhexafluorophosphate, and iron protoporphyrin IX in acetonitrile solution at scan rate of 100 mV/s (Materials and Methods 2.D)

Discussion

1. UV-Vis Spectroscopy

The purpose of this experiment was to analyze the electrochemical properties of various metal protoporphyrins in comparison to iron protoporphyrin IX. Iron protoporphyrin IX, also known as heme b, is a tetrapyrrole located inside the protein hemoglobin. This heme has known reductive properties, acting as an electron-transfer site that, in hemoglobin, effectively transports oxygen through the body's vascular system. The ability of iron protoporphyrin IX to effectively reduce carbon dioxide has been proven in previous studies, and this experiment sought to explore whether the same function could be carried out within a modified protein, and by other metal

protophyrins. Mn, Co, Cu, and Ni protoporphyrin IX were also tested for their electrochemical properties and reductive potential. These properties were examined first through a UV-Vis spectrometer measuring the absorbance of the solution, and then with a cyclic voltammeter to measure the reduction and oxidation potentials of the solution, as well as the reversibility of the solution. Nickel protoporphyrin was found to have the highest level of absorbance, 3.425, at the maximum wavelength when examined through a UV-Vis spectrometer, followed by copper, iron, cobalt, and finally manganese. For the cyclic voltammetry, only solutions of the standard ferrocene aldehyde and iron protoporphyrin IX were tested, allowing for future experiments. Neither test was reversible at both 25 mV/s and 100 mV/s, although the iron protoporphyrin IX did show reductive capabilities at 0.51 V and 0.61 V, respectively.

The first set of UV-Vis solutions, prepared with 2.5 mg of the respective metal porphyrin and 10 mL of DMF, proved too concentrated to examine further. Thus, a 10% dilution of each solution was prepared, with more useful results. Because DMF absorbs light at <270 nm, the results for each UV-vis spectroscopy should only be regarded above 270 nm, as anything less is altered by the solvent. Each metal protoporphyrin showed a maximum wavelength at approximately 400 nm, which shows a bathochromic shift from the standard maximum wavelength of C=C chromophores of 171 nm. This shift is caused by an increased number of conjugated pi bonds and delocalization. The absorbance region of metal protoporphyrins falls in the violet region of the visible spectrum, a value demonstrated by the molecules' distinct purple coloring. While the UV-Vis results did reveal information as to how well each metal protoporphyrin absorbed light energy at certain wavelength, the results could not be compared, as each metal had a different molar capacity for absorption. In order to correct

for this, the molar absorptivity was found for each solution using Beer's Law, which measures the intrinsic absorption value of each metal protoporphyrin. Nickel protoporphyrin had the highest molar absorptivity, and was thus chosen for further examination under UV-Vis spectroscopy.

Nickel dilutions of various concentrations were run under UV-Vis spectroscopy with varying results. The 4.2×10^{-6} M solution had a similar absorbance level and molar absorptivity, 3.5 and $1.00 \times 10^5 \text{ L mol}^{-1} \text{ cm}^{-1}$, respectively, as the previous test, confirming results. The molar absorptivity at maximum wavelength varied from solution to solution. As an intrinsic value, each solution should have had the same molar absorptivity. Aberrations in data could be attributed to the values themselves: because the absorbance is being divided by the concentration, errors in data with such small molarities are amplified, skewing the data.

The addition of the ascorbic acid to the Ni protoporphyrin solutions intended to decrease the absorption to less than 1.0 for more accurate results. Ascorbic acid has been shown in previous studies to cause a hypochromic shift in UV-Vis spectroscopy results, and the same shift occurred in this experiment. All solutions experienced a hypochromic shift at the maximum wavelength, particularly the 4.2×10^{-6} M solution, which shifted from 3.79 to 0.36.

During one trial of nickel dilutions, a new UV-Vis spectrometer was tested that measured transmitted light intensity instead of absorbance. In order to compare the two trials effectively, the new UV-Vis data was transformed in order to give the absorbance of the solutions as well. The data showed to be irregular, with negative absorbance levels outside of the DMF absorption region for the 2.1×10^{-7} M solution, as well as a level of absorbance that leveled off at 0.1 instead of falling to 0.0 for the 3.86×10^{-8} M solution.

These differences in data can be attributed to the equipment, as the same solutions were used for both UV-Vis spectrometers.

2. Cyclic Voltammetry

Cyclic voltammetry was performed in order to determine the energy needed for running a reaction that will effectively reduce the solution, as well as determining whether the catalyst is consumed during the reaction, a negative outcome of the experiment. The first solution tested was one of ferrocene aldehyde and t-butylhexafluorophosphate, a standard solution known to produce successful voltammograms. At both a scan rate of 25 mV/s and 100 mV/s, the solution did not solve for either reversibility equation, signaling that the catalyst was consumed during the reaction. Because this solution is a known reference solution, these results point to an error in the set-up of either the solutions of the equipment. While the equations did not show reversibility, the trends in the graphs did: The current increased with the increasing scan rates while holding a similar cathodic peak potential, a sign of reversibility. The addition of Fe protoporphyrin IX held similar results, with the reversibility equations not holding at both scan rates, but the graphs showing quasi-reversible behavior. As the scan rate increased, the cathodic peak potential increased from 0.51 V to 0.61 V, and the corresponding cathodic peak current increased from 5.87×10^{-5} A to 1.1×10^{-4} A. While some of the results remain unclear and must be explored further, each set of data revealed a consistent cathodic peak potential, giving insight into a starting point for reduction reactions involving metal protoporphyrins in the future.

3. Preparation Work

In order to perform the UV-Vis spectroscopy and cyclic voltammetry experiments, certain compounds had to be synthesized and purified. The first was iron protoporphyrin IX

to be used in the cyclic voltammetry experiments. The percent yield was 48%, showing either an incomplete purification or a very crude beginning sample. Nickel protoporphyrin was also synthesized in preparation for cyclic voltammetry. After the solution of refluxed DMF, protoporphyrin IX, and nickelous sulfate was produced, the solution was tested for red fluorescence. Red fluorescence denotes a presence of pure protoporphyrin, and suggests that more nickelous sulfate be added to the solution. The solution yielded green fluorescence, signifying the necessary hypsochromic shift. Due to time constraints, the complete synthesis of nickel protoporphyrin IX was not attained. The last compound to be recrystallized was t-butylhexafluorophosphate from tetra-n-butylammounium chloride and ammonium hexfluorophosphate. T-butylhexafluorophosphate acts as an electrolyte in the cyclic voltammetry solution. The recrystallization yielded 62%, showing a loss of product. While the original experiment called for 0.2 millimoles of each solute, the procedure was performed with 2.0 millimoles of each solute in order to produce enough of the product to successfully make the acetonitrile solution for the cyclic voltammetry experiments.

4. Errors, Improvements, and Future Experiments

Due to the time frame of the study, completed results could not be attained, and further experiments and improvements to the methods are necessary. The primary improvement would be more extensive testing of the cyclic voltammetry experiments. While effective data was produced, the equipment used was brand new, and more tests must be run in order to gain familiarity with the methods and materials. Improvements could be made by allowing more time for the solutes to dissolve in the acetonitrile, as well as for nitrogen to be dissolved in the system. Not all of the t-butylhexafluorophosphate dissolved in the solution during the first trial, thus

decreasing the electron-transfer ability of the solution. Further studies in cyclic voltammetry and the ability of metal protoporphyrins to reduce carbon dioxide would include metal protoporphyrins besides iron, as well as testing the energies of these molecules inside of proteins, the eventual goal of the research. While a reference electrode of 0.1 M acetonitrile and silver nitrate was used, other solutions could be tested, as discussed in the introduction.

Improvements could also be made to the UV-Vis spectroscopy portion of the experiment. While nickel protoporphyrin IX proved to have the highest molar absorptivity of the solutions, future studies might include dilutions of the other metal protoporphyrins. Repeat trials of the nickel dilutions might also confirm the molar absorptivity and help in explaining inconsistencies in the data. More experimentation on the new UV-Vis spectrometer, which yielded inconsistent and incongruent results, might also lead to insight as to the absorptivity of these metal protoporphyrin solutions. Lastly, because the ascorbic acid was so successful at lowering the absorbance of the nickel protoporphyrin, thus yielding more accurate results, ascorbic acid should be added to every solution as part of future experiments.

The low percent yields of the purified and synthesized compounds point to errors in the procedure. During the purification of iron hemin, not all of the protoporphyrin went into the solvent. This limited the amount of purified hemin that could be produced, and improvements could be made by allowing for more time for the solute to enter solution, or by exploring different solvents besides pyridine. Systematic errors occurred during the synthesis of nickel protoporphyrin IX that rendered the sample unusable. Improvements could be made by stabilizing the reflux equipment in order to ensure that the nickel protoporphyrin can be produced without lab hazards. The low percent yield of the t-

butylhexafluorophosphate could also have occurred due to a systematic error of a surplus of ethanol being used during the recrystallization. The excess ethanol slowed down the process, and also overwhelmed the Buchner funnel, causing precipitate to fall through into the beaker. This resulted in a loss of mass, and improvements could be made by dissolving the crystals in less ethanol.

Conclusions

This study of metal protoporphyrins revealed important information regarding the electrochemical properties of the samples and how they might be used to reduce carbon dioxide inside a modified protein. The UV-Vis spectroscopy results showed iron-, cobalt-, manganese-, copper-, and nickel-protoporphyrin IX solutions to absorb maximum light energy at approximately 400 nm, providing valuable information as to how light energy might be effectively used for reduction reactions. The data served as a starting point for future experiments, with nickel protoporphyrin having the highest molar absorptivity. The cyclic voltammetry experiments, while inconclusive due to time constraints, showed quasi-reversibility for iron protoporphyrin IX solutions, pointing to room for future experiments with the other metal protoporphyrins.

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**She Blinded Me With Science:
Boiling Acetic Acid Outside of the
Fume Hood and Other Beeghly
Mishaps and Musings**

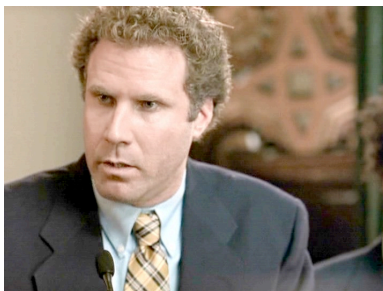
A man walks into the bar and is shot. The paramedics rush to the scene, and, seeing that he is losing blood quickly, ask him, "What happened? Who did this to you?" The man replies, "Heme! It was heme!"

1.

There's No First-Person in Science, but Quite a Bit in This Capstone

Dear Advisors, Mom, Dad, bored and/or hungry Honors students waiting in line for Cake Day¹, and maybe a handful of friends I bribe with Super Mugs to edit this,

Congratulations on making it through the first part of this capstone project! I just finished writing it myself, and I honestly don't know how I did it. I feel like Will Ferrell after he delivers his genius, inspired debate speech in Old School. "What happened? I blacked out." In fact, this was my exact expression after I finished my report:



Complete and total mental shutdown. In case you didn't get around to reading it, I will provide you with a summary: for my Honors capstone project, I spent a semester working as a research assistant with Dr. Hartings at the AU Chemistry Department. The research involved the modification of hemoglobin, and the molecules, known as hemes, inside of them. Heme is particularly effective at electron transport, and the purpose of this research was to identify how these hemes reduce carbon dioxide both inside and outside of hemoglobin, and then to test other metal protoporphyrins to compare their reductive potential. It was just as fancy as it sounds. Dr. Hartings even constructed a glove box and cyclic voltammetry station just for the occasion:



I couldn't resist running it through Instagram. Vintage! Anyways, this capstone seemed like the natural next step in my growing interest in chemistry, a field I hoped to pursue in graduate school, and I believed serious lab work to be a bridge from the humanities to the hard sciences. You see, I am an unlikely science student. My parents are both microbiologists, and while I now find the "we met at the microscopes" story adorable and endearing, their insistence that I not begin my thermodynamics lab reports with two-page, verbose expositions on the more poetic aspects of taking one's morning coffee became fodder for rebellion

¹ Viva la Cake Day, Cake Day forever

when I was World's Most Obnoxious Preteen²: if my parents were scientists, then I was going to be anything but. This immaturity was coupled with terrible science teachers in middle school and high school, whose answers to my struggles (I still don't understand why an object will hit the ground at the same time regardless of whether it is dropped or given a horizontal trajectory. Confusion, thy name is physics) ranged from "you're bad at science because you suffer from low self-esteem" (an astute but absolutely unhelpful and potentially sexist response) to "Sarah, don't go into science." Great, thanks. I came to AU as a philosophy major, which I think about every time I'm having a bad day and need a good laugh, and later declared my major in environmental studies when I realized that I will never care about anything more than I do learning how our world works and how we might protect it. I had considered going academically green before, but was turned off by all of the science that it would require. During the second semester of my freshman year, I bit the proverbial bullet and walked into the hallowed halls of Beeghly for the first time for General Chemistry I; just like in a romantic comedy, when the just-friend shows up on your doorstep in the middle of the thunderstorm holding a dozen roses and you suddenly realize that you've loved them all along, I too fell in love with science.

I loved everything about chemistry. There was something incredibly satisfying about learning exactly how the world works. That's a pretty hyperbolic statement for an introductory class, but I suddenly felt that all of the answers- both the small and large ones- were within my reach, if I just read my textbook and did the legwork. Doing Mastering Chemistry assignments in the library at 2 a.m. suddenly became a Zen exercise, where excelling at a class meant more answers were solved, not raised. This does not, of course, mean that I did not recognize the natural curiosity inherent in any science. This curiosity is driven by facts, not feelings, and chemistry became a solid landing on which to place my feet while navigating the often-depressing, always-confusing waters of my environmental policy and development classes. In those classes, I was told that the world was going to end; science taught me why this was happening and how it could be stopped. I was drawn to not only the factual nature of chemistry, but also to the beauty lying behind it; not only did I now have an insight into how the world worked, but that insight was remarkably ordered and awesome in its mechanisms. I continued to take chemistry courses throughout my time at AU, and felt that working in a lab, my first semi-independent foray into scientific research, for my Honor Capstone project would cement that experience.

What I did not account for in my quest to be a super scientist (a quick search for this job title yields no LinkedIn results, but more Café Press t-shirt and coffee mug results than I'm comfortable with) is that I am, and have always been, a complete and total scatterbrain. I have absolutely no attention to detail. My catchphrase in high school was "close enough." I'm an awful parallel parker not because I don't have the skills to do it correctly, but because I often just don't care to take the time to fix the 45° angle at which I've parked my car. I avoid unpacking for weeks because I'm terrified of having to follow the directions for assembling IKEA furniture. One time, I tried to put together a chair, and it immediately collapsed. It was traumatizing. I just sat there in shock in my dorm room, clutching its scattered remains in my trembling fists. This head-in-the-clouds syndrome paired with my natural clumsiness makes me a recipe for a lab disaster. I am a bull in a china shop if that bull were often texting while walking and trying to accomplish basic daily tasks. When F. Scott Fitzgerald talked of the "careless people" in *The Great Gatsby* who "smashed up things and creatures and then retreated back into their money of their vast carelessness," he was actually talking about me, and the money he's referring to is Eaglebucks. That is a little known literature trivia nugget. You're welcome! My picture for Dr. Hartings' lab website should have been a tip-off:

² Winner of the title of World's Most Obnoxious Preteen at the 2002 American Angst Awards



I don't even look like a real scientist. *I popped the collar of my lab coat, for Pete's sake.* I look like someone cast Zooey Deschanel in a movie where she plays a lovable zoologist with adorable quirks who falls in love with the man who wants to shut down the zoo and direct the funds to a sweatshop, or something. You know, pave paradise and put up a parking lot. I had to make her a zoologist because there's no way Hollywood would allow a female lead in a romantic comedy (I am aware that this is my second reference to rom-coms, and I'm okay with that) to be an analytic chemist. There must be scenes of her studying adorable lion cubs, not cyclic voltammeters. Electrochemistry is intimidating, and who will love us then? Which brings me to my next point:

I want you to picture a scientist in your head. Got it? I'm thinking of my high school physics professor, a meek, balding, pot-bellied man who wore plaid short-sleeved button-up shirts and was rumored to have a trophy-wife. Even as part of the choir to which I am preaching, my mental image of science-minded folks is still straight out of *Big Bang Theory*. And that's not fair, because we're a pretty cool bunch.

Most people assume that because scientists have unemotional, technical jobs regarding inaccessible subject matter, that they are unemotional, technical, inaccessible people. And that's not true at all! The science professors at AU have been some of the funniest, warmest, most interesting people I've met at this school. They make jokes about Bolsheviks and Cheez Whiz, they listen to David Bowie and Mad Men Spotify stations with us in the lab, they show us pictures of their pets in class and open up to us in times of personal strife. This disconnect is mildly comical when in sitcom form (seriously, why does everyone think *Big Bang Theory* is so funny?), but dangerous when encountered in the real world.

It only takes a political ad or a newspaper article to realize the disrespect that scientists encounter, and how hard facts have been disregarded for mere ideology. The recent contraception debates pin fact against opinion, with conservatives attacking what is medically deemed as preventative medicine, but what they view as an "abortion pill," stripping women of power of their own bodies. This separation is particularly strong in environmentalism. Scientists have become the enemies of climate change deniers, the latter taking information that should stay in the lab and taking it to the floor of Congress. Even among environmentalists, the role of technology is heavily debated and extremely contentious. Behaviorists believe that if you tell enough people to love Mother Nature and use a refillable bottle, that all of these monolithic problems, issues that have ultimately changed the very biological, geological, and chemical cycles and systems of the Earth, will suddenly disappear, as if *The Lorax* were a work of nonfiction. I find myself among the rare breed of environmentalists who hold that a problem with scientific roots needs a scientific fix. Our technological and intellectual progress- along with a healthy dose of respect- is the only cure we have for a very ill Earth, and this advancement will only happen if we begin to make science and math education a priority, ending this notion that black-and-white scientific truths can be dyed to match Red or Blue states. It is not just an issue among conservatives, either: if I hear one more person rally against genetically modified organisms or nuclear

power without any knowledge of how these technologies actually work other than “they’re bad,” I am going to hang myself with a reusable tote bag, I swear to god. We must change the misconception that scientists are somehow removed from and aloof to the concerns of the public, bopping around the lab without a care as to the implications of their research. The truth is that they care very much.

For my 20th birthday, I proposed having a chemistry-themed party where everyone would dress up as their favorite element and enjoy chemistry-themed decorations and libations. I tried to explain to my roommate how I wanted to go as fluorine, the most electronegative element. I told her that fluorine is the element that steals everyone else’s electrons, and, as such, I was going to dress up as a mean, popular girl who steals your boyfriend, a modern-day interpretation of the element. I have never seen a blander or more judgmental stare. I never did have that party, but maybe after this capstone, there will be a whole new crop of people (okay, five people, max) with the understanding that there’s room for humor in the lab. Guys, science can be fun, I promise! Chemists have feelings too, even if we do check them at the lab door with our food and drink. I may not have been great at using a UV-Vis spectrometer, but hopefully I can make you laugh a little bit. Why so cerium?

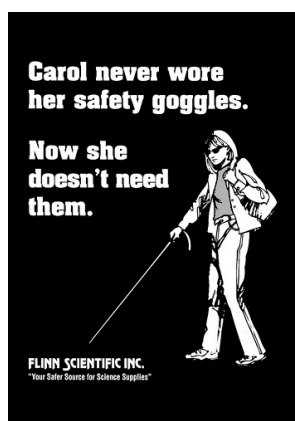
2. The Danger Zone

The lab is a dangerous place. I know this both from experience and tall tale. Every chemistry student at AU is required to watch a lab safety video during the first class of every semester. I don't remember the name of the video, but I'm sure it's something along the lines of "Mind Your Crop Top!: How to Stay Safe in the Lab in the Mid-90s." Although I have sat through this video three times now, and know exactly what is going to happen, it is still a horrifying experience, scary in both its subject matter and tackiness. The video opens in a generic-looking chemistry lab full of actors who appear to be way too excited about being in lab. No one in the history of anyone anywhere has ever shown even the slightest inkling of enthusiasm about lab. Ever. I don't care if you're a future Nobel Prize winner; you still dread the lab portion of your chemistry class. Lab is a three-hour ordeal, a class you want to be over as quickly as possible, only to realize that as soon as it's finished, you are that much closer to the due date of your next lab report, an assignment that guarantees an all-night library panic session, fists manically clutched to a coffee and a Red Bull as you frenetically flip through the pages of your organic chemistry textbook trying to figure out what a Zaitsev product is, and where the hell those extra electrons on the mechanism go. To this day, I'm still not sure.

While independent lab research is personally fulfilling, overseeing the experiments that you purposefully designed with your research interests in mind, lab coursework feels removed, with each week being an isolated lesson that holds little connection to previous labs or lectures. Certain lab techniques must be mastered, and labs are an ideal way to familiarize students with complicated procedures. What labs lack, however, is that gratifying feeling of discovery for discovery's sake. The end product is the grade, the A with the perfect data. In reality, the data is not always completely congruent, and being able to perform a semester's worth of research for an ongoing study with results relevant to the real world was completely rewarding, seeing the experimental aspect of chemistry as more of a pet project and less of a pet peeve.

Anyways, the point is that these young adults in the safety video look like they're enjoying lab to the point that we begin to grow concerned about their mental stability, the first unsettling part of the video. The second involves the dangers that await the students as they are picked off one by one like some sort of sick chemistry version of The Hunger Games. Someone leans over the counter too far and catches on fire from an ignited Bunsen burner. Another victim splashes chemicals in their eyes, crying over an eye rinsing station as an omniscient narrator describes the pain and permanent eye damage that occurs when corrosive chemicals are trapped behind contact lenses. The most terrorizing scene is one involving glassware, in which a student unwisely tries to wedge a tube into a rubber stopper, hitting resistance and instead puncturing the glass shards into his hand, blood spewing down his arm as the tube juts out like a violent Tower of Pisa. As someone who is on "Faint Watch" (I'm not sure if this is a technical term, or if they had to make it up just for me) at the doctor's office whenever there is blood work or needles involved, this mental image did not sit well with me. As a Gen Chem student, I imagined every lab ending in a trip to the hospital, my body riddled with glass fragments, hair (and Scrunchie! Again, this was made in the 90s) burnt to the scalp, skin melting off from rogue acid reactions of fellow careless students. As previously discussed, I am not the most graceful of people, and the safety video made the lab seem like a klutz's most hellish nightmare realized: a place where beakers explode and natural gas threatens to ignite at any given moment.

After weeks passed in the lab, though, and no disaster struck, experiments limited to mixing simple solutions and running them through chromatography columns, I grew complacent. Surely this video was a lesson in histrionics, the chemistry equivalent of a middle school "family life" lecture that preaches that every sexual encounter will end in both pregnancy and HIV. I was also reminded of the infamous cautionary poster that graces the walls of every high school science classroom:



Poor Carol. It was not until beginning my capstone research that I realized that what made independent research away from a classroom setting so great was also what made it that much more dangerous.

When doing research as part of an independent study, as this capstone entailed, there are no waivers to be signed or safety videos to be viewed; it is expected that those working in the lab have sufficient experience to understand the chemicals and equipment involved, and that, given this added responsibility and autonomy, they will handle these potential hazards with care. With the experiments being designed by those who will perform them, there is a wider range of chemicals being utilized, some innocuous and some more dangerous. I remember one day in particular involving the purification of iron protoporphyrin IX using chloroform as a solvent. I knew chloroform wasn't a substance that you could just casually throw around, but a quick Wikipedia search revealed a bevy of hazards: chloroform is not only a suspected carcinogen, but it can cause immediate death due to cardiac arrest when as little as 10 mL is ingested. The substance was used for some time as an anesthetic before it was proven to cause sudden cardiac arrhythmia in patients, and it has also been linked to miscarriages in rats. Even dastardlier, chloroform has an instant depressive effect on the nervous system when inhaled, and this property has been harnessed in order to daze or kill. The use of chloroform as a sedating agent has been linked to robberies, sexual assaults, and murders. Every warning on every label of every bottle held the same advice: "DO NOT SWALLOW. DO NOT INHALE. DO NOT TOUCH. THIS STUFF WILL SERIOUSLY...MESS YOU UP." I'm not sure what level of profanity is appropriate in an Honors capstone, but you get the picture. The severity and gravity of lab safety finally set in as I poured chloroform into boiling acetic acid, blood-red iron protoporphyrin IX stains splattered over the lab station (how meta), and realized that not only did my station look like a crime scene, but it could actually be a crime scene if I tried. Or, in my case, didn't try.

While I loved the self-sufficiency that an independent lab study allowed, I will admit that I missed the metaphorical crutches of a lab class. Knowing my penchant for accidents, spills, and other assorted mayhem that comes from inattention to one's surroundings and long, awkward limbs, I was *that person* in lab who made sure to have the T.A. beside them at all times. I was a shameless T.A.'s pet, constantly pestering them to double-check that I was obtaining correct data, and, more importantly, was not going to blow up anything or cause a Beeghly ruckus. This capstone kicked the crutches out from under me, took off the training wheels from my chemistry bike, threw me in the pool without water wings³. Without a lab partner, I was left to my own devices, and the results were not pretty. These were the unsavory errors not included in the lab report:

³ In case you haven't noticed, I tend to speak in hyperboles, and this is not exception. Dr. Hartings was an extremely helpful and accessible advisor, stressing lab safety and encouraging questions from Day 1. The difference lies in a class where the goal is to Do It Right with prescribed procedures v. a research experience that promotes the scientific process and exploration where the student has some freedom in the design of the project.

The aforementioned purification of iron protoporphyrin IX had a percent yield of 48%, showing a loss of product. Possible errors that could have caused this loss of mass included the systematic error of improperly dissolving the protoporphyrin in the pyridine. Possible errors that could have caused pulmonary edema included me boiling acetic acid outside of the fume hood. This occurred during the second trial of the experiment, and I had been previously instructed not only to boil the acetic acid under the fume hood, but to keep my skin away from the fumes when stirring in other compounds. Prepared to boil the acetic acid, I found the heating plate under the fume hood to already be in use. “Instead of waiting for this reaction to be finished, I’ll take matters into my own hands and find another heating plate,” I thought to myself. “No need to bother Dr. Hartings!” This plan obviously backfired. As I waited for the acetic acid to come to a boil, ~~texting~~ using my time productively to wash beakers and begin next week’s prep work, Dr. Hartings took one whiff of the room:

“What smells like vinegar?” He looked around nervously. “Sarah, where is the acetic acid?”

“It’s on the heating plate on the counter! I decided to do some preemptive prep work for the purification,” I beamed, oh-so-proud of myself for being a proactive, productive chemist.

This, of course, was not the correct response. My embarrassment was roughly on par with when, on my first day of lab, I reached for a pencil in my backpack but only had a pink Hello Kitty pen, except this instance held a higher risk of respiratory failure. Much like I developed a liquids-around-electronics neurosis after spilling a beer on my boyfriend’s computer and completely destroying it, I now take special care never to take a substance out from under the fume hood. Does sodium chloride need to be massed? Tough cookies, it can’t leave the fume hood. Dihydrogen monoxide, you sit your ass back down, do not even *think* about leaving that fume hood.

The second “human error” might have been my biggest screw-up to date. Like, ever, not just in the chemistry lab. The incident occurred during the synthesis of nickel protoporphyrin IX, a molecule later to be analyzed via cyclic voltammetry. I walked into lab already distracted that day; the AU Career Fair was later that afternoon, leaving me preoccupied with my lack of networking skills, the discomfort of my heels and pantyhose, and general uncertainty of post-grad life, with graduate school admissions letters still looming. The synthesis procedure called for a reflux of the solvent DMF; in order to reach the exact temperature, water is run in plastic tubing from the lab station through the reaction, and then back behind the lab station into some sort of receptacle. The experiment involved a lot of waiting, and as I massed nickelous sulfate in preparation for the next step, I began to hear running water. I knew that the reflux reaction was occurring, and didn’t give much thought to the noise. Several minutes later, I turned around to find the entire lab station flooded. The floor, the counter, everything, just completely covered in water. The force of the water must have propelled the tubing out from behind the lab station, creating a hose that showed absolutely no mercy.

I have never in my life experienced such panic as I did in that moment. While I try to take things lightly and find a comical perspective to every situation, I am completely serious when I say that I am incredibly, fantastically, unbelievably lucky and fortunate that no one was hurt during the lab that day. Water is certainly a more innocuous substance to have spilled over your entire lab station than chloroform or something more hazardous, but water is still reactive with many chemicals (the beloved mantra of “add acid” comes to mind, as well as the YouTube videos that Dr. Hirzy loved to show of water being added to cesium), and could have caused serious damage. As I scanned the deluge, I also noticed that three appliances were currently plugged in at the time. These appliances were fortunately mounted on an insulating plate, thus protecting them from the onslaught of water. Had this not been the miraculous case, true disaster could have ensued, the water sparking an electrical fire that could reach the highly reactive chemicals on and under the lab hood. I cringe merely at the thought of what might have happened in a less forgiving universe. In addition to the chemical dangers, the water also managed to spread across a ten-foot radius around the lab station, creating a slipping hazard for anyone walking in the lab. I am far from religious, but I can’t help but think that

I had a little guardian chemist angel on my shoulder that day, making sure that no one was hurt as a result of the accident. “She’s too young to die! She hasn’t even started her capstone yet. There are so many puns she hasn’t made yet!”

I will be following a less chemistry-intensive academic path at grad school, which is perhaps for the best for those who would otherwise be stuck in a lab with me. This semester’s lab research has taught me many valuable lessons, one of which I’m pretty sure I once read on a Yogi Tea label: you can’t change who you are. I can’t change that I love learning how the physical world around me works, I can’t change that I take a nerdish glee in solving chemical equations, and I can’t change that I will drop 57% of the objects that I pick up. And that’s ok! Our skill sets and interests won’t always intersect in a perfect Venn diagram. The second lesson is that the lab safety video holds many universal truths that must not be ignored. Acid will be boiled. Lab stations will be flooded. Chemical waste will be mistaken for dimethylformamide and poured into beakers. Be vigilant. Be aware. Don’t be Carol.

3. Escape from Azkaban

Much like Lindsay Lohan in *Mean Girls*, in which she has a theory that “If you cut off all of Regina’s hair, she looks like a British man,” I too have several theories. The first is that when TDR is out of silverware and dishes due to a, air-quote, broken dishwasher, that the dishwasher is not actually broken. The second is that Fake Neil Kerwin of Twitter fame is actually Neil Kerwin. The third is that one to several of the men I have dated have not been completely straight. Among these deeply held conspiracies is the belief that Beeghly is actually Azkaban.

For those of you not familiar with the term, Azkaban is the fictional prison of the Harry Potter series, reserved for the foulest of magical criminals. It is set on an island in the middle of the sea, completely inescapable to those who inhabit it. Azkaban was once guarded by Dementors, creatures that feed on human unhappiness and exist solely to make you as miserable as possible. The mere sight of one causes despair, depression, and utter devastation. The Dementor’s Kiss leaves its victim without a soul, a shell of a former person. If you would like more information on Azkaban or Dementors, there are entire articles devoted to the subject on Harry Potter Wiki, a website obviously written by people who do not have a capstone due in six ~~days~~ hours.

I’ve made it clear that I have loved my chemistry classes. I really do. I might be the only sick bastard who has taken organic chemistry as an elective. But, like any good relationship, not every day can be roses and puppies and marshmallow sunshine kisses. Most days I strut into Beeghly, kicking my feet up on a swivel chair as I excitedly await the beginning of lecture, or popping the collar on my lab jacket as I prep for the day’s experiments. In the words of Soulja Boy, I do, in fact, have my swag on. There are other days, however, where the walk to Beeghly feels like a death sentence; it is the days when you are in a fight with your significant other and you forgot to pay the gas bill and you have five assignments due and you haven’t slept more than three hours the entire week and you are having an existential crisis that the windowless, colorless interior of Beeghly becomes a prison. I had one of those days almost every day last semester. I remember bumping into a friend on the Quad one day; she must have noticed my bloodshot eyes and vacant stare, my arms weakly grasping at my organic chemistry textbook, and she asked where I had been. “Azkaban,” I flatly replied. “I’ve been in Azkaban.”

What makes Beeghly so akin to Azkaban is its location. Just as part of the punishment of Azkaban is its isolated setting on a rock in the ocean, hundreds of miles from any other human contact with only the endless water around you and no hope for escape, Beeghly too is located as remotely as possible from the rest of campus. When you have over seven hours of classes there in one day alone, you are truly stranded. Removed from the hustle and bustle of the Quad and MGC, there is a very real separation from the rest of campus that is almost tangible after a long day. You cannot simply pop outside for a second and hope to see a friendly face, to essentially press the reset button on a stressful day. Unlike every other building at AU, there is no real lobby or meeting space in Beeghly. While students come and go throughout the day, the dearth of communal areas makes the building seem as if you are the only person there, an eerie, unsettling feeling that is not made any better by the narrow hallways and tiled floors. And, just as Azkaban finds itself separated by water, going to or from Beeghly in the rain is a miserable, soul-sucking experience. On certain days, it is enough to make you curl up on a bench outside the lab and not even leave the prison at all.

The draining, empty feeling so distinctly characterized by Azkaban comes to a head when you realize that lunchtime is approaching, or you have a 10-minute break from your evening block class, and there is neither food nor coffee to be found. Beeghly may be the only building at AU without access to caffeine, with

the exception of Hurst, which is still a two-minute walk from The Day. There is something so hopeless, so soul-wrenchingly desperate about the idea of not being able to grab a cup of coffee during a block class, or give yourself a quick caffeine jolt before bunkering down in the lab. As someone who once habitually consumed eight cups of coffee a day, and can drink an oversized mug of coffee at midnight and be in bed an hour later, having a steady stream of caffeine before handling hazardous materials and following extremely detailed, technical procedures is an absolute necessity. One lab experiment in organic chemistry entailed an extraction of caffeine from tea, and the tension within the students over whether or not to consume the coveted substance in its purest, most powerful form was palpable.

I made the mistake once of studying in Beeghly at night. In the midst of finals week, I knew that outlets would be few and far between in the library, and, seeking a quiet study space with minimal interruptions, I ventured into Beeghly. What is draining during the day becomes terrifying at night, the building being transformed into the set of a horror film. As you walk down the dimly lit, narrow basement hallways, fluorescent lights buzzing in an empty, hollow monotone, the complete vacancy of the building and your isolation from campus becoming increasingly apparent, it is not hard for your sleep-deprived, caffeine-shocked brain to start imagining mad scientists in blood-soaked lab coats lumbering down the hallway, filled with rage after being trapped in Beeghly à la *Jane Eyre*. Suddenly, your imagination overpowers your need for an outlet, and the threat of becoming a character in the next Saw movie sends you sprinting from the building in a panic-stricken flight. It is an escape from Azkaban.

Beeghly has the privilege of being the only building at American University neither scheduled nor budgeted for renovations in the foreseeable future. Due to the hazardous nature of the building's contents, renovation costs have been estimated at \$50 million⁴. Beeghly is well overdue for a complete revamping, but several smaller changes could be added in order to turn it from Azkaban to Hogwarts. Simply adding more color to the interior and increasing the amount of natural or natural-looking light would make the building more inviting, particularly in the basement. The most important addition, however, would be the creation of a communal student space. The most popular buildings on campus offer not only classroom space, but areas conducive to studying and socializing as well. Even the older buildings on campus, such as McKinley and Hurst, have comfortable couches and chairs in their lobbies, suggesting that the student is welcome even when class is over. A few seating options in the first floor lobby of Beeghly, along with a few outlets, would instantly transform the space. While the most drastic of changes, the renovation of the second-floor study room into a student-run coffee lounge, much like The Day, would drastically improve the atmosphere of Beeghly. Students would not be forced to leave between their classes, or sit on the floor of the hallway while finishing up problem sets before class; instead, a coffee lounge, named something like Cesium the Day, The Coff-man Product, or Van der Waals, would create a community of chemistry students and faculty, making the trek from the Quad worth it. In *Harry Potter*, wizards and witches can vanquish a Dementor with a Patronus, a summoned spirit that chases away the soul-eaters. While we cannot rebuild Azkaban anytime soon, we may be able to cast enough Patronus spells to make Beeghly just a bit more magical.

⁴ According to a conversation I once had with Dr. Cheh

4. Not Everyone Can Do Science...

As graduation approaches, the conversation among us seniors has inevitably turned to just what exactly we are going to do with the beginning of our adult lives. While I have been emotionally and mentally prepared to leave college for a long time, what those who have gone before us failed to mention is the shock not from the transition itself, but from the suddenness at which we are plunged into the real world⁵. One day you are shamelessly bringing your laundry to your parents' house, perusing their pantry for snacks to take back to your temporary home of Craigslist furniture and coffee mugs that double as wine glasses, and the next day you are a veritable grown-up with grown-up responsibilities like finding a job and eating three meals a day that aren't coffee-based and figuring out what the hell a mortgage/equity loan/[insert finance term here] is. As is the trend for the fine members of Generation Y, I have decided to delay adulthood for as long as possible, and instead of "making an income" and "investing in bonds" and "buying appropriate work clothes" (that last one was my mom's idea) I will be furthering my education at Montana State University next fall. I'm so excited! I will be earning my Master of Science in Health and Human Development, Snowboarding, and Trying to Be Interested in State School Sports but Probably Never Going to a Single Game. Just kidding, the last two are minors. What I will not be studying, and what is noticeably absent from that list given the subject matter of this capstone, is chemistry.

Not pursuing a graduate degree in a chemistry-related field is certainly not from a lack of trying. Last semester, I applied to four graduate schools, hoping to continue my research in protoporphyrins and metalloproteins. While I did not expect to get into every school, my 25% success rate, only being accepted into Montana State, stung a bit. I know that I sound bratty, whining about how I didn't get into my top choices for grad school as I sit in the library of a private university, essentially smothered in privilege, but these circumstances did not cushion the blow of rejection. When you apply to a job, there is only so much that is invested in the process. Submitting only a resume and cover letter, perhaps the same documents you've sent to dozens of other employers, you know that the rejection comes from someone in HR who scanned your resume for six seconds and threw it away. The application process for graduate school, though, is so much more extensive and personal. You spend weeks huddled over a GRE prep book, write draft upon draft of your personal statement, invest countless hours researching graduate schools and their programs, build rapport with advisors and begin to plan your field of research, and stay in a constant state of existential crisis as you mull over what you really do want in life, and whether it can be attained from a graduate degree. When you finally do decide where you want your life to take you, and that a particular program can get you there, the rejection letter is that much more painful. I consider myself pretty thick-skinned, but when my parents called to tell me a small envelope arrived in the mail from University of Oregon, my top choice, I cried. It is the same pain of unrequited love, of investing in a relationship only to be told that, no, you are not wanted. As rejection letters arrived from University of Oregon, UC Davis, and University of British Columbia, I shook my fists in the general direction of the west coast: "You don't know my life! YOU DON'T KNOW ME AT ALL."

I approached the graduate school process with another dangerous Generation Y mentality, that, if you try hard, put your best foot forward, and have a good attitude, then success will come, and that the act of making an effort is, and always should be, rewarded. This approach has won me countless "participation ribbons" for coming in last place at swimming competitions (my asthma and complete lack of competitive drive make me a terrible athlete). It earned me ice cream and hugs from my parents when I did poorly on a

⁵ Not to be confused with the hit MTV reality show, "The Real World," although if that is your post-grad plan, who am I to judge?

test but still “tried my best.” It gave me a bloated sense of confidence and security that I think I share with many people my age. It did not, however, get me into a graduate chemistry program.

The hard sciences may be the only field that, no matter how badly you want it, no matter how qualified you are⁶, no matter how much you devote yourself to your research, is sometimes just completely unattainable, and has a cap as to the number of people that can participate. Not everyone can do science. All graduate programs are selective to some point, and this is not to imply that those studying the non-sciences have walk-on graduate programs. The difference lies in funding. While there is certainly a cap to other programs, at the end of the day you will be paying for a spot, and if you are qualified, intelligent, passionate, and financially able, the world is your academic oyster. Most openings for science programs at graduate schools, however, are extremely limited by the funding granted for each department, or even individual advisor, who is then allowed to handpick one or two students to be accepted into the program, adding another personal dimension to an already subjective system. The schools and programs are not to blame for this, but the funding surely shapes the nature of the graduate school process. My rejection letter from University of Oregon stated that the department only *had eight funded spots* open that year for over 90 applicants. That acceptance rate is lower than the current unemployment rate. No, it's okay, I feel dead inside, too.

These circumstances allow admissions committees to be hyper-selective in hilarious ways. The application for UC Davis's Agricultural and Environmental Chemistry program asked for a list of your inventions, which marked the exact moment at which I knew I was royally screwed. The only thing I have ever invented is choffee. Choffee is a scrumptious beverage prepared by making instant hot chocolate with coffee instead of water or milk. Developed out of sheer necessity late one night in the dorms, it is truly brilliant in its simplicity, but unfortunately, I do not think that the admissions committee at UC Davis was ready to experience that level of genius and innovation just yet. “By Jove! This choffee is the samizdat of beverages,” they would exclaim, carefully alluding to the fact that they read *Infinite Jest*. My other invention, though never brought to fruition, is a cashmere-lined bra that I will sell at my lingerie store, Abreasted Development. That said, I left that box blank on the application.

The catch with not being able to pursue the hard sciences in an academic setting is that you cannot turn them into hobbies. Perhaps it's possible if you have an endless supply of free time and disposable income, like an elderly, eccentric heir to a financial firm or hotel chain, but the average Joe and Jane Doe cannot perform cyclic voltammetry experiments at their home on leisurely weekends. You cannot join the Neighborhood Metalloproteins Association, or casually fire up your UV-Vis spectrometer during your free time. The best you can do is visit your local science museum and order a subscription to *Science*. The world of chemistry becomes completely inaccessible without a formal education, an education that is difficult to attain in the first place.

It is likely that I am writing this essay from a place of bitterness; if I had been admitted to UC Davis or University of Oregon, I would probably be wearing a lab coat right now (yes, in the AU library), pushing my glasses up the bridge of my nose and nodding my head in smug, patronizing agreement. “Ah, yes, not *all of us* can do science.” I am beyond excited to begin my research at Montana State University, examining the nutritional value and sustainability of community food security programs while working towards becoming a Registered Dietician; ensuring that people have healthy relationships with their food and with their Earth is an issue close to my heart, and I am chomping at the bit to start this new field of study. Truth be told, though, I am going to miss chemistry. I will be taking nutrition courses at MSU that are rooted in biochemistry, but my chance to study chemistry as a pure science, to feel that same fascination I experienced in Dr. Hirzy's class three years ago over and over again as I go through graduate school, doctorate work, post-doctorate research, and eventually professional studies, is gone. While many interests can be picked up later in life, pursuing chemistry would require starting over again at an undergraduate level; perhaps what

⁶ Okay, so I wasn't that qualified

triggered my emotional response to University of Oregon's decision was not the rejection by the school, but the idea that the door leading to something that I love has been closed, a realization that has been slowly dawning on me over the course of this semester. That's okay, though. Not everyone can do science. I will be doing something else that I love, something that will improve the lives of others, something that will get me out of bed every morning during the cold Montana winters. I will leave the chemistry to someone else, someone who lives and breathes their research, someone who has invented something other than a less-classy café mocha. I am sure that there are more than eight intelligent, ambitious people in this world with not only big ideas, but also the knowledge to turn those ideas into a reality.

5. ...But Everyone Should

My four years at American University can be summarized by the following pie chart:

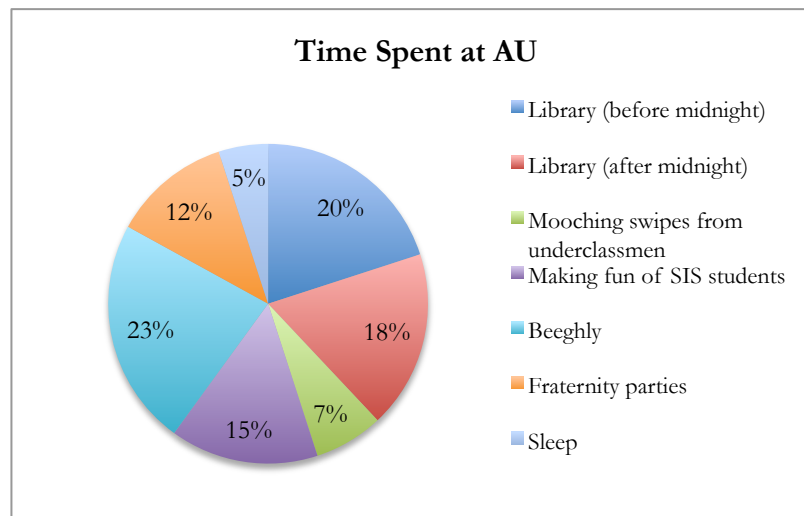


Chart 1. How I spent a \$160,000 education, approximately

As you can see, 15 percent of my time here has been spent making snide remarks about SIS students, just slightly more time than was spent at fraternity parties, but not quite as much as was spent panicking in the library after midnight. My mockery comes not from a place of disrespect, but of sheer laziness. There are over 3,000 students in the School of International Service, a third of total AU enrollment; combine this statistic with the vocal, politically minded nature of the student body, and it's only a matter of time before you find a Global Environmental Politics grad student who feels the need to inform your 500-level Environmental Science II class that China's carbon footprint is rising, or an undergrad recently returned from studying abroad who politely corrects you on your pronunciation of Chile. "*It's CHEE-leh.*" It is like shooting fish in a barrel after discussing international trading rights and quota policies. This gentle teasing is done out of love; I would much rather see an international relations program full of idealistic, passionate people than militaristic neoconservatives. Still, what really gets my goose, more so than any recurring conversation at a party about the state of the euro, is the perfunctory dismissal of science by SIS students, both as a legitimate field of study at AU, as well as its relevancy to their research.

American University is almost defined by its students' disconnect from math and science. The departments themselves are strong, but it is hard to work with a group of people who openly complain that Group 5 of the AU General Education program, the physical sciences, should not be required, and is completely unnecessary for a well-rounded education. This separation from and ignorance of science creates a double standard across the schools. Occasionally, this double standard works in my favor. Nothing garners more sympathy during finals week than telling people that you're working on a chemistry assignment.

"Man, that sure is a heavy stack of books. What are you writing your paper on?"

“I’m writing a 40-page paper on the commoditization of culture following a post-imperial era of globalization and BRIC microloans for rural development⁷. Also, sex trafficking. You?”

“I have a lab report to finish up.”

“Oh my god, that sounds *awful*. You poor thing.”

It’s fun to trick people into thinking you’re smarter than you really are, but the double standard, by definition, goes both ways. The above conversation usually ends like this:

“Yeah, it’s pretty rough. I just got back from a really long day in Beeghly. Kinda want to die.”

“...Beeghly?”

“Yeah, Beeghly. You know, the chem building?”

“We have a chem building? Where? Does that even exist?”

Beeghly does in fact exist, for those of you who were wondering, and it is located behind Centennial Hall, next to the Southside student parking garage. It’s okay, though, not all of us can have brand new LEED-certified buildings that serve as a selling point of the university. It’s fine, I understand your confusion.

What I do not understand, however, is the prevailing attitude at AU, particularly among SIS students, that one is expected to have an extensive knowledge of international relations regardless of one’s field of study, while at the same time refusing to take a science class seriously, laughing off one’s lack of knowledge about the subject. When I do not have an articulate, conclusive opinion on how to resolve the Israeli-Palestinian conflict, it is because I am ignorant and uninformed; when someone is in tears over a BIO-100 lab, it is because “science is hard,” and for whatever reason, is not worth learning.

On the contrary, science is applicable to all subjects, and a basic background in physics, chemistry, and biology can enhance one’s education and reveal new ways of looking at the world. While I would consider myself a science student, I am technically graduating with a B.A. in Environmental Studies. This program requires classes in both the physical and social sciences, and I do not believe that my education would have been complete without the latter. An understanding of the physical world is essential, but without a means of communicating that information, a way of understanding of how society affects and is affected by these factors, or a process for invoking change in order to best utilize scientific discoveries, this knowledge can only be so useful. My science education was greatly enhanced by international relations, politics, economics, and sociology, and the reverse holds true as well. Going back to the Israel-Palestinian conflict, how can you discuss conflicts driven by the limited availability of water if you cannot explain how the water cycle works, or the science behind the climate change that is leading to both an increase in water-stressed and flooded regions? How can you debate the future of nuclear conflict in the region if you do not understand the uranium enrichment process?

SIS and CAS may be separate schools, but they study the same world viewed through different academic lenses. Just as everyone should have a working knowledge of international events and the theories and social forces surrounding them, everyone should have familiarity with basic scientific concepts. For better or worse, science and policy have become inextricably linked, intertwined to a point that ignoring one for the other is not only irresponsible, but also reckless. The physicist unaware of policy unwittingly creates the atomic bomb; the politician uninformed of physics agrees to launch it.

⁷ I don’t know what this means