Method Development for Analysis of Dietary Supplements Using Flame Atomic Absorption Spectroscopy

Elijah Hongsermeier-Baxter and Jennifer Firestine
Lindenwood University School of Chemistry, Saint Charles, Missouri

Abstract

Dietary supplements are typically ingested by customers who already have a well-balanced diet and an awareness of a personal, specific deficiency or fitness goal serving as their reason for doing so. Accordingly, the accuracy of the composition of a supplement on its label is paramount in determining if said tablets are worthwhile for customer use. A method was developed to determine the accuracy of the nutrition facts labels on two different brands of iron and zinc supplements. Samples were prepared via dissolution in nitric acid followed by filtration, dilution and analysis via flame atomic absorption spectroscopy. A standard curve method was used. Results of experimental trials were analyzed against the standard curves developed and results were compared to the manufacturers' labels.

Introduction

Two commercially readily available brands of dietary iron and zinc supplements (Nature's Bounty and CVS Pharmacy) were analyzed for accuracy of mineral concentrations. Highly accurate nutrient labels are key when it comes to supplements, as consumers typically buy these nutritional products with a specific fitness objective or nutritional deficiency in mind. Incorrect labels can be misleading and cause consumers a false sense of security. According to Carol Haggans of the Office of Dietary Supplements (ODS), "It's possible to get all of the nutrients you need by eating a variety of healthy foods, so you don't have to take one."* This suggests that the desired audience of this product primarily includes individuals who are in fact more serious/mindful about their health and fitness than the average citizen for any variety of reasons. These include ambitious fitness goals, restricted diets due to allergies, natural deficiencies of specific nutrients, or disease rehabilitation.

Calculation Title	Formula	Result
0.5 ppm Standard	1000 ppm std/0.5 ppm std	dilution factor: 2000
1 ppm Standard	1000 ppm std/1 ppm std	dilution factor: 1000
General Standards	1000 ppm std/x ppm stds	dilution factor: 2000-200
50 mg Supp. Dilution	500 mg L^(-1)/2.5 ppm std	dilution factor: 200
65 mg Supp. Dilution	650 mg L^(-1)/2.5 ppm std	dilution factor: 250

Table 1. Preliminary calculations performed before procedure

Procedure

1. A tablet containing 50 milligrams (mg) of iron (Fe) was placed in a beaker along with 40 milliliters (mL) of 3 molar HNO_3 (nitric acid) for 20 minutes to dissolve.
2. A serial dilution was performed on a 1000 ppm Fe standard down into 0-5 ppm standards in 0.5 ppm increments.
3. Solution from step 1, after digestion, was gravity filtered into a 100 mL beaker using a glass funnel and filter paper.
4. Resulting solution was poured into 100 mL flask along with water to fill line, then diluted down to a 2.5 ppm solution in a new flask with water.
5. Standards from steps 2 were ran through flame atomic absorption (FAA) spectrophotometer to develop a standard curve, followed by the sample completed in step 4.
6. Steps 1-5 were repeated for CVS Pharmacy brand Fe supplement.
7. Steps 1-6 were repeated for Nature's Bounty and CVS Pharmacy brand Zinc (Zn) in the place of Fe and 1000 ppm Zn standard in the place of Fe standard, as well as a 650 to 2.5 ppm dilution (dilution factor of 250) rather than 500 to 2.5 (factor of 200).
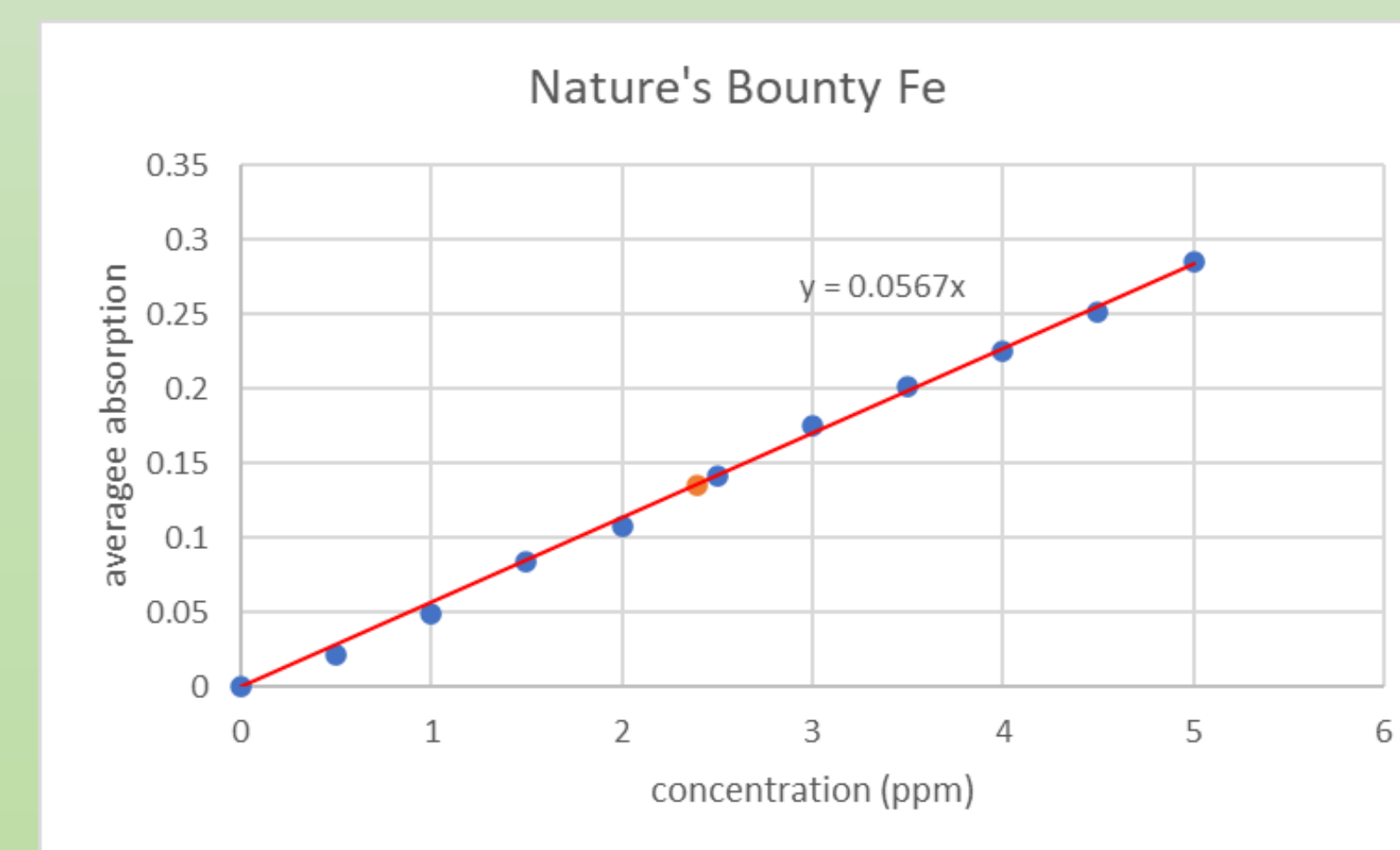

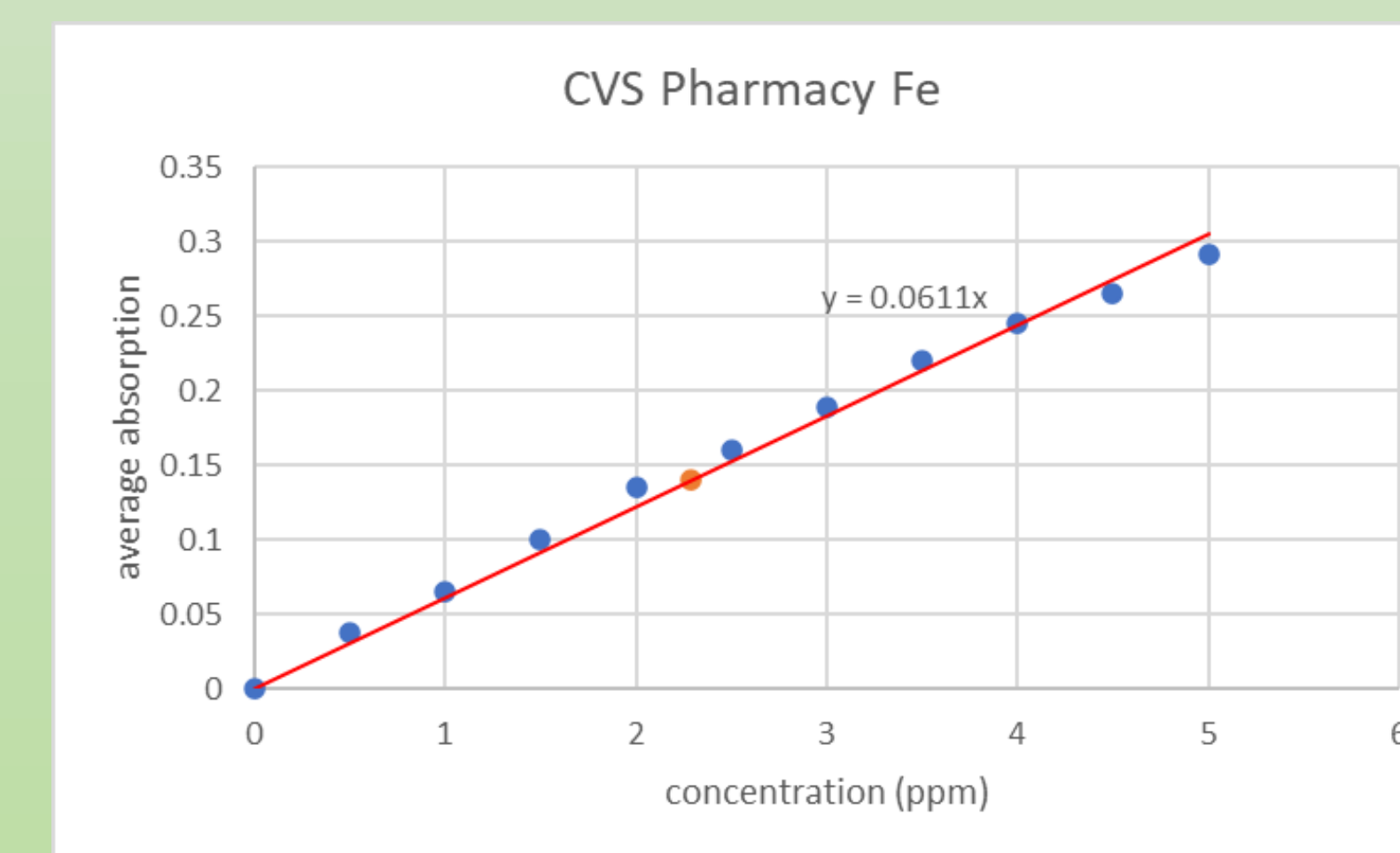*Chart 1. Nature's Bounty iron sample plotted against standard curve*

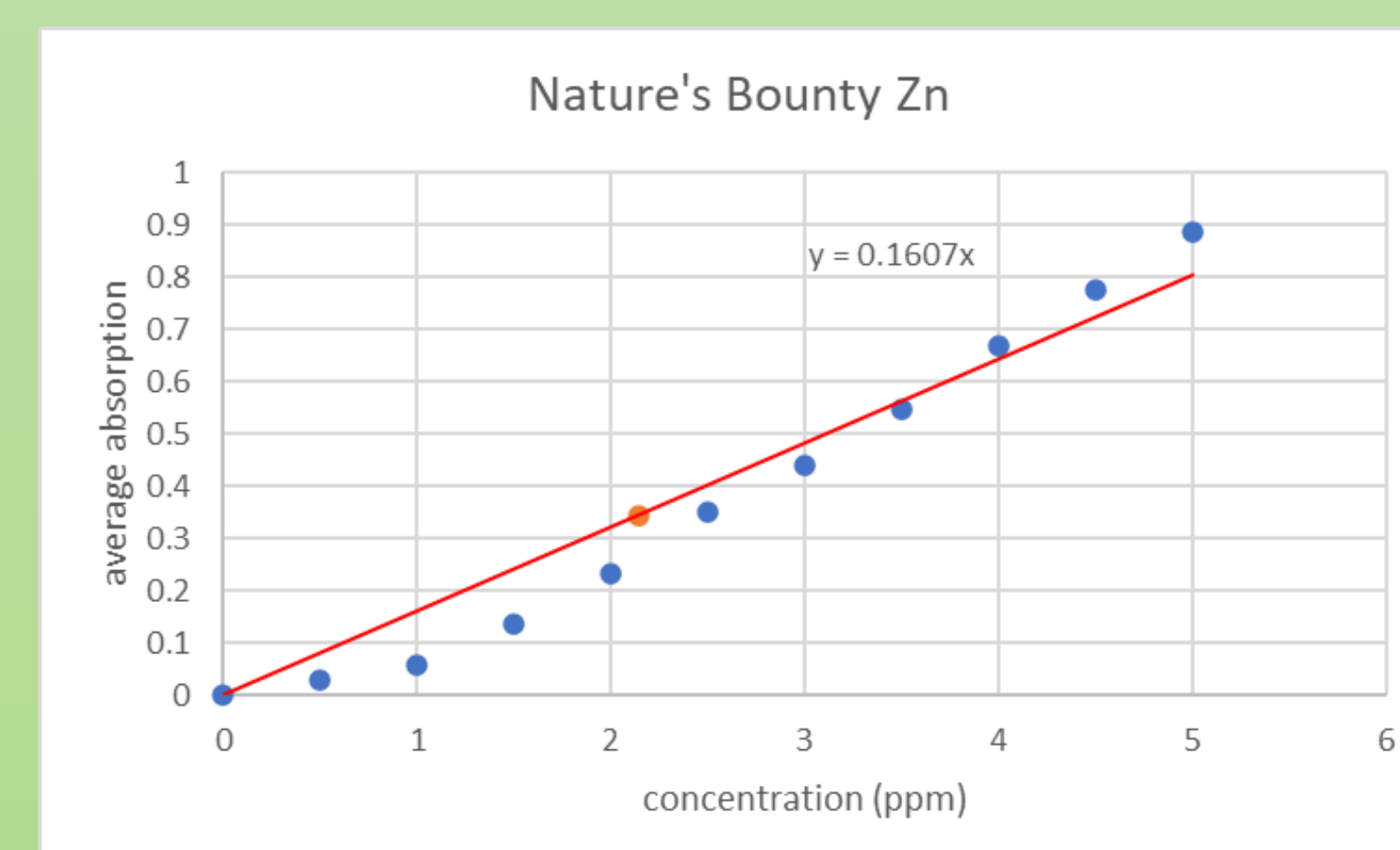*Chart 2. CVS Pharmacy iron sample plotted against standard curve*

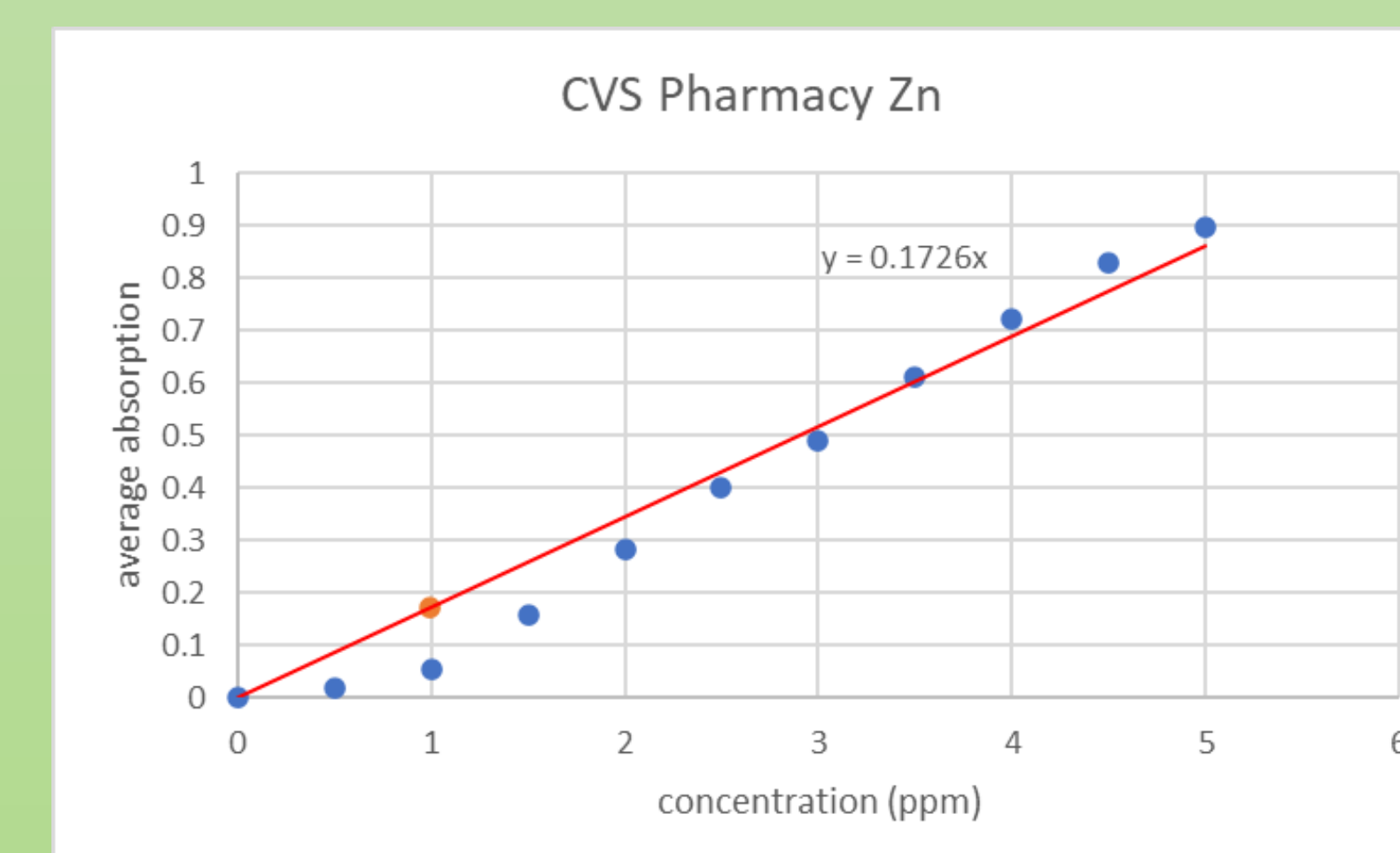*Chart 3. Nature's Bounty Zinc sample plotted against standard curve*

Chart 4. CVS Pharmacy Zinc sample plotted against standard curve

Title	Formula	Result
Sample (y=mx+b)	x=(y-b)/m	x
NB Fe Concentration	x=0.136/0.0567	2.399 ppm
CVS Fe Concentration	x=0.14/0.0611	2.291 ppm
NB Zn Concentration	x=0.345/0.1607	2.147 ppm
CVS Zn Concentration	x=0.172/0.1726	0.997 ppm
CVS Zn in HCl Concentration	x=0.115/0.1606	0.716 ppm

Table 2. Calculations performed after procedure and data collection to determine results quantitatively

Figure 1. Example photograph of gravity filtration

Figure 2. Photograph of a PerkinElmer AAnalyst 200 (instrument used)

Results

Referring to Charts 1-5 and Table 2, a trend is clearly noticeable that Nature's Bounty was more accurately labelled than CVS Pharmacy products. Samples containing Nature's Bounty's iron and zinc supplements came in at concentrations of 2.399 ppm and 2.147 ppm respectively, compared to a theoretical concentration of 2.5. The samples containing CVS Pharmacy iron and zinc supplements came in at just 2.291 ppm and 0.997 ppm respectively, compared to a theoretical value of 2.5 ppm. One final trial was conducted, substituting HCl for nitric acid to recreate the environment of the human stomach to ensure acid choice was not the source of error, which resulted in an even lower concentration of 0.716 ppm. This said, such noteworthy discrepancies between theoretical and experimental concentrations for CVS Pharmacy samples may be the result of a number of errors. One possibility is that there was error in the dissolution process, resulting in some amount of zinc not making it through the filter paper and into the sample. Another possible source of error was the samples themselves, which may not have had uniform distributions of minerals in diluted acid throughout, causing misreads. Lastly, the instrument itself may have been inconsistent or inaccurate, which could lead to misreads as well. Conducting more trials and gathering more data would help mitigate these errors.

Conclusions/Future Work

Based on the results gathered from the five trials to the left, it is clear that Nature's Bounty does a better job in providing value to customers through dietary supplement tablets than CVS Pharmacy for only a slight increase in price. The fifth and final trial (pictured below) was conducted for the sole purpose of ensuring the massive discrepancy in theoretical and experimental concentrations from trial 4 would carry over and directly apply in the human stomach. The next step would be to repeat trials 1-3 in HCl as well to validate findings. Following that, branching out to test the concentration of other vitamins and minerals in these same brands to further validate the overall trend would support above claims even more. A good supplement to begin with would be Magnesium. Clearly, there are many other directions in which this work could be directed in theory, but the final one I will include here is, after thoroughly supporting claims about the accuracy of Nature's Bounty and CVS Pharmacy nutritional labels, branching out to test the accuracy of nutritional labels on the product lines of different brands, such as NatureMade, Kirkland, OLLY, etc.
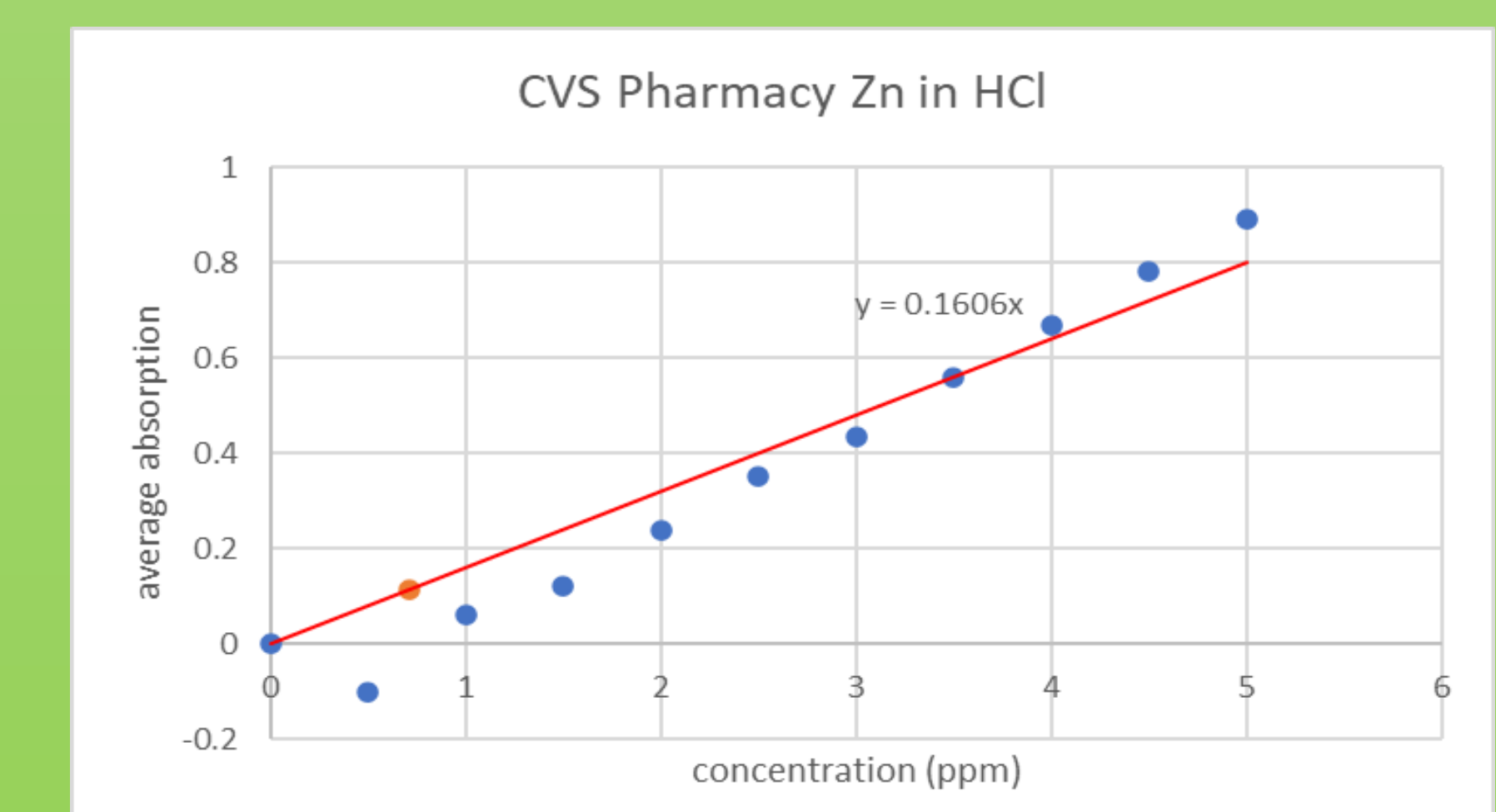

Table 5. CVS Pharmacy Zinc sample dissolved in HCl plotted against standard curve

Sources

News in Health Home Page. https://newsinhealth.nih.gov/2013/08/should-you-take-dietary-supplements/ (Accessed 2022-03-01)

Harper College Page on Gravity Filtrations. http://dept.harpercollege.edu/chemistry/chm/100/gdodambe/thedisk/labtech/filter2.htm/ (Accessed 2022-03-01)

American Laboratory Trading Atomic Absorption Spectrometer Page. https://americanlaboratorytrading.com/lab-equipment-products/perkinelmer-aanalyst-200-atomic-absorption-spectrometer_11371/ (Accessed 2022-03-01)