Analysis of Cu and Sulfate Concentration in Multivitamins

Names of Authors

Introduction

In this experiment, the purpose was to find the Cu concentration in multivitamins and to see if it was stable over time. This was done using a Flame Atomic Absorption Spectrometer (FAAS) and a Ion Chromatograph (IC).

A FAAS is useful for determining a concentration of a specific metal in a sample by using the cations. It is very useful for detecting metallic elements with accuracy, precise quantitation, and within very sensitive detection limits. The reason this works is because atoms of each element have a specific electron configuration for their ground state and when these atoms absorb energy they will emit light that has a specific wavelength that will correspond to the allowed transitions within the atom.[1] The solution with the sample is aerosolized into a acetylene flame when it is atomized and the color of the flame will inform what wavelengths of light the sample absorbed. In this experiment the purpose was to determine the concentration of copper in a multivitamin and to compare this result to the amount of copper reported in that multivitamin. As a group we determined that the amount of copper in the multivitamin tablet would be similar to the amount reported by the company.

The IC seperated and quantifies ions in a solution using the mobile (sodium carbonate/bicarbonate eluent) and stationary phases (column packed with resin beads to give a positive charge). The IC is used in this experiment to determine the sulfate concentration in the multivitamin tablet. According to the label of the multivitamins, they do contain sulfate but the supplemental facts does not give us an amount of sulfate contained in these multivitamins. However, on the list of ingredients the sulfate is listed as ‘cupric sulfate’ so the amount of copper in the multivitamin should correspond with the amount of sulfate. As a group we hypothesised that the copper and sulfate concentrations should be extremely similar, if not the same, due to them both being added as part of the same compound to the multivitamin.

Materials and Methods

Sample preparation

Comment [AF1]: You did a decent job describing the techniques used, but what should the reader know about the analyte? Why would Cu be important?

Comment [AF2]: Good reference format

Comment [AF3]: Good thought.

Comment [AF4]: This entire section reads like a list of orders/instructions. It should instead be a past-tense account of what you did.

This section should include a summary of what you did in lab and not a step-by-step recitation of the procedure. This applies to the entire section, but as an example you could just say “The multivitamin was sonicated in ultrapure water for 5 minutes, diluted 1:10, and filtered through a 0.45 µm filter prior to analysis.” You don’t need to say what size vol flasks you used, how much you pipetted, how many times you transferred to a new container, etc. Someone else may do all of those things differently based on the glassware they have available. The details listed here are important in your lab notebook, but you should summarize them when preparing your manuscript.

In this experiment, there were two different instruments being used, so therefore two different sample preparations happened. The two instruments are the Flame Atomic Absorption (Flame AA) and the Ion Chromatography (IC).

Flame AA

For the standard preparation, a stock solution of 10 ppm Cu SASS (standard addition spike solution) was provided in this lab. Then, place 10 mL of the stock solution in a 100 mL volumetric flask and to bring the volume of the flask with 5% nitric acid.

For the sample preparation, place a multivitamin pill in a 10 mL volumetric flask with 50 mL ultrapure water, sonicate mixture for 5 minutes, bring to volume with ultrapure water, then transfer to container. Take a 100 mL volumetric flask and put 10 mL of precious solution of the multivitamin and dilute it to 100 mL with 5% nitric acid. Then, filter 70 mL of that with a 0.45 micrometer syringe filter (dispose of the first 5 mL of it). Take six 25mL volumetric flask and add 10 mL of the sample to each flask, then add 0, 1, 2, 3, 4, and 10 mL of the 10 ppm CU SASS to a flask, and label the flask with appropriate volume added. Bring all six flasks to volume with 5% nitric acid and transfer samples to a 15 mL conical vials.

IC

For the blank preparation, use ultrapure water.

For the standard preparation, a stock solution was prepared by using 4.0792 mg of Sodium Sulfate and 200 mL of ultrapure which made a concentration of 20 ppm. The stock solution was used to make four other concentrations through dilution. For each four solutions made from the 20 ppm stock, take 10, 5, 1, and 0.5 mL of the stock and dilute it with 20 mL of ultrapure water to get 10, 5, 1, and 0.5 ppm.

For the sample preparation, take a 100 mL volumetric flask put a multivitamin pill and 50 mL of ultrapure water in a flask, sonicate for 5 minutes, fill volume to 100 mL, and transfer to a container. Take 10 mL of the sample and put it in a 100 mL volumetric flask and dilute water to volume. Filter 10 mL of sample with 0.45 micrometer syringe filter (disposing the first 5 mL).

Instruments

When the standard and sample preparations are done, it is time to run the instruments. Each instrument has a set of guidelines that should be followed at the instrument. Flame AA is performing a absorption measurement using a Copper hollow-cathode lamp. The wavelength that was used was 324.75. The IC is using Sodium Carbonate/ Sodium Bicarbonate as the eluent with a flow rate of 1.2 mL/min and it is injecting the samples at a volume of 5 microliters. Below is the parameter for the IC.

Table 1: IC method

Comment [AF5]: This actually was the stock, not the SASS. You all made the SASS in lab.

Comment [AF6]: Yes, it is.

Comment [AF7]: Again, this level of detail belongs in your lab notebook. You can say "a 20 ppm stock solution was prepared with sodium sulfate in ultrapure water".

Comment [AF8]: Make sure to reference the table by number.

Comment [AF9]: This should be on the same page as the table.

Parameter	Value
Mobile Phase	Carbonate/Bicarbonate
Ion-exchange Group	Alkanol Quaternary Ammonium
Construction Material	Peek
Flow Rate Range	1.2 to 2.5 mL/min
Diameter (mm)	4mm
Length (m)	150 m
Cost ($,US)	$1,194.47

Comment [AF10]: Would be nice to include the concentration here.

Comment [AF11]: This should be the flow rate you actually used.

Comment [AF12]: Careful with units!

Comment [AF13]: This table should include all the experimental parameters that someone needs to know to repeat the study. The cost isn't necessary for someone to know who is trying to reproduce your study.

Results and Discussion

	A_S_Loc	Sample_ID	Date	Time	Analyte_Name	Elem	Wavelength	Abs_Corr_	SD_Corr_Abs_	RSD_Corr_Abs_
	Int64	String	String	String	String	String	Float64	Float64	Float64	Float64
1	1	Calibration standard	3/4/2019	9:00:06 AM	Cu 324.75	Cu	324.75	0.0139537	0.000517136	3.7061
2	2	0 mL	3/4/2019	9:00:18 AM	Cu 324.75	Cu	324.75	-0.0141844	0.00142344	10.0353
3	3	1mL	3/4/2019	9:00:35 AM	Cu 324.75	Cu	324.75	0.000232395	0.00731792	3148.91
4	4	2mL	3/4/2019	9:00:51 AM	Cu 324.75	Cu	324.75	0.0224545	0.0115281	51.34
5	5	3mL	3/4/2019	9:01:08 AM	Cu 324.75	Cu	324.75	0.0409073	0.0134559	32.8936
6	6	4mL	3/4/2019	9:01:24 AM	Cu 324.75	Cu	324.75	0.058929	0.0161877	27.4697
7	7	10mL	3/4/2019	9:01:42 AM	Cu 324.75	Cu	324.75	0.168109	0.0368439	21.9166

Figure 1: Raw Data collected using Flame Atomic Absorption in order to find the Cu concentration

Comment [AF14]: This table should be cleaned up a little. The reader doesn't need to know that your samples were run at 9 AM or what AS location the vials were in. Only include information essential to the story about your results – the rest should be in your lab notebook.

Comment [AF15]: This is a table, not a figure.

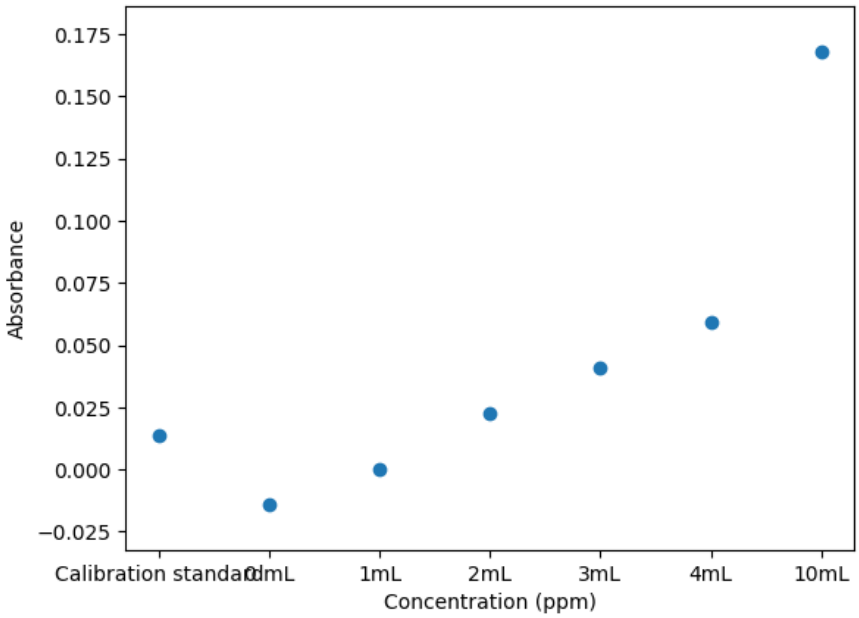

Figure 2: Scatter Plot including the Calibration Standard of FAAS

Comment [AF16]: The x-axis is labelled as concentration/ppm, but the values show ml. Milliliter is not a measure of concentration. Also note that the spacing is the same between each data point – you plotted the x-axis as categories (strings), not continuous numbers.

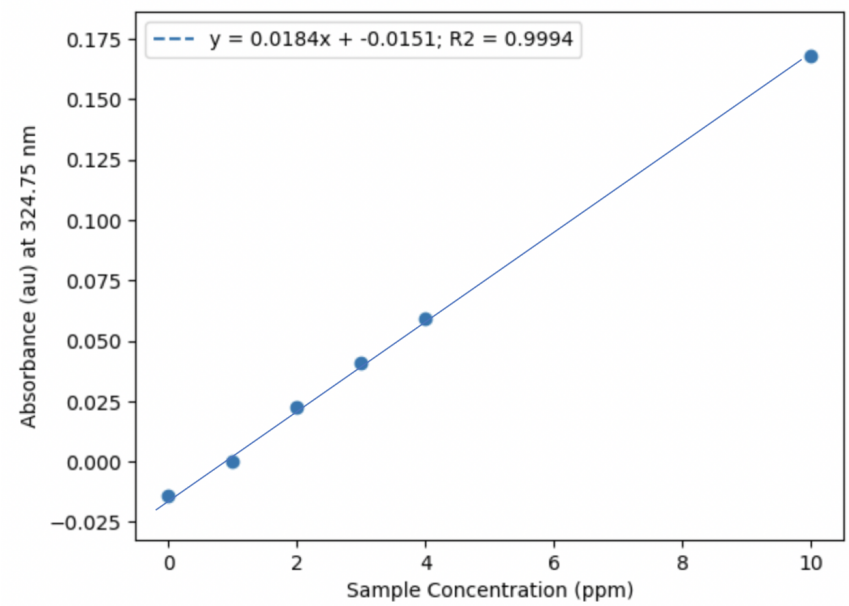

Figure 3: Spectra including line of best fit and reported R value.

The Flame Atomic Absorption was used in order to determine the amount of copper found to be in the multivitamin pill provided. Through this process it was would that found the amount of Copper in the multivitamin was approximately 0.8206 mg. According to the container in which the multivitamin came in, the amount of copper listed should have been approximately 9 mg. This gave us a 90.88 percent error rate and while this only approximately 0.08 mg difference it is still over 5%. There are numerous reasons as to why this might be. This could be a slight error in which the company that makes the multivitamin pills made. During the last 4 steps of the sample preparation we were supposed to add 10 mL of our sample to 25 mL flasks, then add the appropriate amount of CuSASS and dilute to volume with nitric acid. The 10 mL and the appropriate amount of CuSASS was added but it was not diluted to volume and was transferred into small vials. However, the mistake was caught, and the solution was transferred to 25 mL vials and diluted to the volume there. This more than likely was the cause of most of our error because we were not able to precisely dilute to volume.

The IC Data was inconclusive, and we had difficulties with the IC instrument. Furthermore, the calculations were based on 25 mL volumetric flasks however they were made in 50 mL volumetric flasks which could have introduced errors into our data. Furthermore, there were issues when saving the data from the instrumentation to a flashdrive in order to analyze the data

Comment [AF17]: This looks somewhat better. Why did you include the same plot twice? This plot still show volume added in ml, although the axis is labelled as concentration in ppm.

Comment [AF18]: Is the x-axis concentration or volume?

Comment [AF19]: What is the error on this measurement?

How did you get this number? Your plot indicates you have a negative concentration…

Comment [AF20]: Not a rate

Comment [AF21]: Isn't it a 9 mg – 0.8206 mg = 8 mg difference (based on the previous sentence)?

using juliabox. The data was not saved as a .csv file, it was saved as a .txt file. The file was able to upload the data onto juliabox, however, it could not separate the columns due to "unrecognizable characters," so it was not possible to create a graph using these files. We were able to download some files off the computer that gave us somewhat of a graph, however the amount of sulfate was unable to be told from this graph.

Comment [AF22]: I still didn't find your IC data submitted.

Conclusions

When looking at the data collected from this experiment we were able to interpret the data from the FAAS but not from the IC due to issues with the data file and how it was saved. When comparing the amount of copper found in the multivitamin through the use of the flame AA to the amount of copper in the multivitamin listed under the supplemental facts on the bottle of multivitamins, we were able to get a concentration with only 0.0794 mg difference. The percent error was over 5% so this was a significant difference, however this could be explained due to some errors when creating the samples. Overall, the method appeared to be successful but there were errors due to transferring the sample between containers. The concentration of the copper found by analysis was close to the amount reported but there was over 5% difference so there is a significant difference. However, if this experiment was repeated with more attention paid to the instructions then the difference between the given amount and the amount found could be made smaller and possible made to be insignificant.

The IC data was not able to be analyzed and so did not produce any data to conclude from. The data needs to be saved into the proper format in order to analyze the data to determine the amount of sulfate in the multivitamin.

References

[1]White, C. Atomic Absorption Determination of Zinc and Copper in a Multivitamin.

Comment [AF23]: Check the ACS format – link on BB.

Data Availability: Where the data are available

CHEM 370 Lab Report Rubric

Vitamin

Grade	Points earned:	10	13	53	29	65
	Group Report Grade (%):			63		

wt.			5 = Excellent, 3 = Average, 1 = Lacking				
			5	4	3	2	1
1	Title	Is the title descriptive and succinct?		x			
1	Introduction	Does the Introduction clearly state the overall question/purpose of the study?	x				
1		Has relevant background information about the analyte of interest been given?					x
1		Has relevant background information about the technique been given?			x		
1		Is there anything that could be omitted?		x			
2	Materials & Methods	Are methods detailed enough that the study can be repeated by another trained scientist?				x	
2		Are there any irrelevant details that could be omitted?					x
2		Are instrument methods clearly detailed, including tables where appropriate?				x	
3	Results & Discussion	Has the main finding been clearly presented?					x
5		Are there factual, logical, analytical, statistical, or mathematical errors?					x
3		Are all figures and tables clearly explained, in order?					x
3		Have the results been related back to the question(s) posed in the Introduction?				x	
5		Is there sufficient data and/or supporting evidence to support the answer?					x
3		Have the results been put into perspective by relating them to literature libraries, standard data sources, and/or expectations? Have errors been provided?			x		
1		Is there anything that could be omitted?					x
3	Conclusions	Has the overall interpretation of the results been clearly conveyed?			x		
3		Are the conclusions free from logical errors?					x
1		Has the study been adequately summarized?				x	
3		Has/have clear conclusion(s) been presented regarding question at hand?					x
1	References	Have references been cited where needed?			x		
1		Are sources cited adequately, appropriately, accurately, and in the ACS format?				x	
1		Are all the citations in the text listed in the References section, and *vice-versa*?	x				
1	General Style	Is the document written according to the CHEM 370 writing guide?			x		
1		Are sections clearly labelled?		x			
1		Are the transitions between sections and paragraphs logical?			x		
1		Are paragraphs and sentences cohesive?			x		
1	Composition	Are there any grammar, punctuation, or spelling errors?			x		
1		Is the style concise?			x		
1		Is there excess wordiness?			x		

Overall Impressions	
What are the manuscript's main strengths? [A]	See below.
What are the manuscript's main weaknesses? [B]	
What specific recommendations can you make regarding revisions to the paper? [see report]	See report.

Adapted from: "Writing Papers in the Biological Sciences" by Angelika H. Hofmann. Oxford University Press 2016 (2nd ed.)

[A] The figures are very good!
[B] There are factual/logical errors in the conclusions drawn.