COVER PAGE, ABSTRACT	**2.0**
RESULTS	**3.0**
ANALYSIS	**3.0**
TOTAL	**8.0/10**

LABORATORY REPORT

GROUP NUMBER: W3

EXPERIMENT NUMBER: 3

TITLE: Atomic Absorption Spectrophotometry

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ROLE ASSIGNMENTS

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ABSTRACT

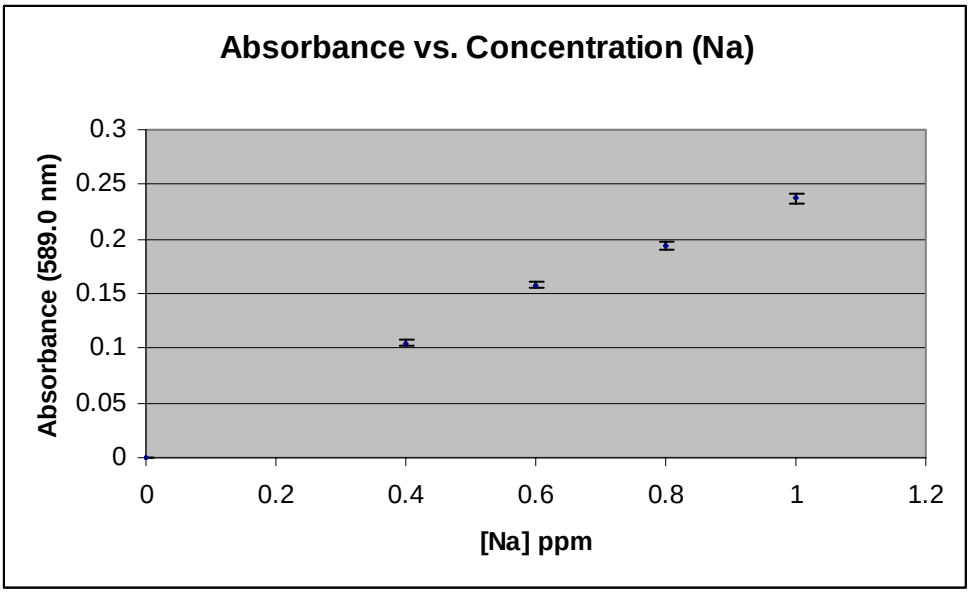
The concentrations of two unknown sodium solutions were investigated using atomic absorption spectrophotometry. The concentrations were determined by taking diluted samples ranging from 20-100% of each of the unknowns and using a spectrophotometer to find their respective absorptions. Concentration values were then calculated using the line-of-best-fit equation, y=0.2371x + 0.006, from the calibration curve of the stock solution. Regression analysis of the stock solution values showed a linear relationship between concentration and absorption (R^2=0.9944), and showed that the y-intercept, 0.006027, was not significantly different than 0 at the 95% confidence interval. The concentration values of unknown 1 and unknown 2 were determined to be

80 ± 2 ppm and 142 ± 3 ppm. These values were in error from the known values by 20.0% and 5.33%, respectively. GOOD ABSTRACT, CONCISE AND TO THE POINT.

RESULTS

The results of the experiment include the absorbance vs. concentration calibration curve for Na, its data analysis, and the obtained concentration values for the two unknowns. These are shown in the two figures and two tables below.

Figure 1. Calibration Curve for standard Na solution



The concentration of the original standard solution is 1000 ppm. The graph above shows that absorbance increases linearly with concentration. The maximum fractional uncertainty is ± 2.1% (THIS IS THE UNCERTAINLY OF THE ABSORBANCE, WHAT ABOUT UNCERTAINLY IN THE CONCENTRATION?).

Figure 2. Calibration Curve with Line of Best Fit

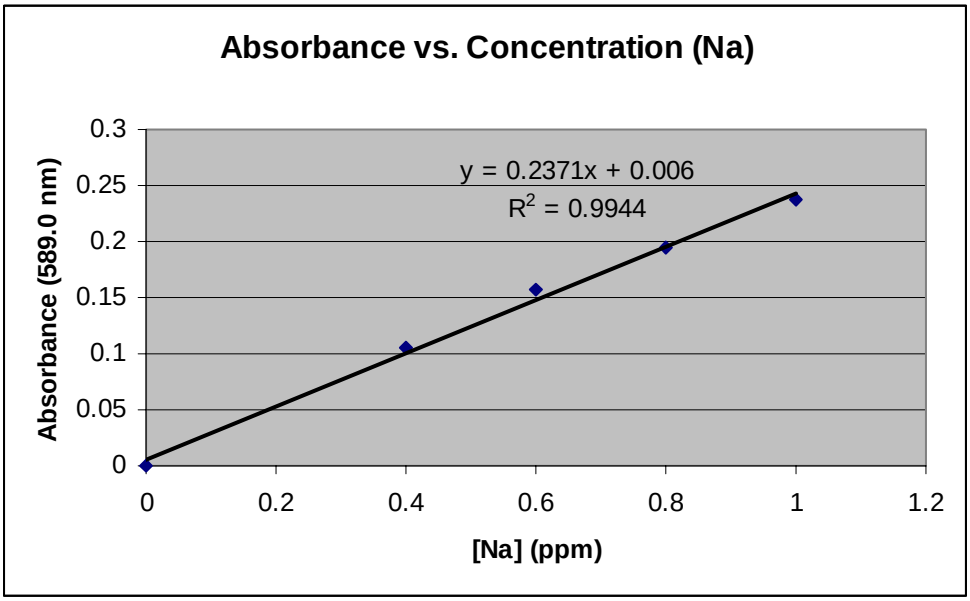


Figure 2 is a reproduction of the calibration curve in Figure 1, with the addition of a linear regression line. THIS IS REDUNDANT, FIGURE 1 COULD HAVE BEEN ELIMINATED TO PRESENT THE SAME RESULTS MORE CONCISELY. IT ALSO APPEARS THAT THE ZERO POINT WAS USED IN THE REGRESSION. YOU SHOULD MENTION THIS. A TABLE WITH THE DATA OBTAINED FROM ACTUAL TRIALS AND AVERAGE VALUES WOULD BE INFORMATIVE. Analysis of this curve in the table below (Table 1) shows that zero lies within the 95% confidence level of the curve, proving the curve significant. A more extensive explanation of the confidence level is described in the caption following Table 1. YOU DO NOT DESCRIBE HOW MANY TRIALS WERE OBTAINED FOR EACH DATA POINT. DID YOU USE THE AVERAGE VALUE OF 3 TRIALS FOR EACH POINT IN THE REGRESSION? DO YOU THINK THAT IF ALL OF THE INDIVIDUAL TRIAL VALUES WERE USED, THERE WOULD BE ANY DIFFERENCE IN THE RESULTING CONFIDENCE LIMITS?

Table 1. Calibration Data Analysis
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.9972
R Square	0.9944
Adjusted R Square	0.9925
Standard Error	0.007921
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	0.03328	0.03328	530.5	0.0001793
Residual	3	0.0001882	6.27E-05		
Total	4	0.03347			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	0.006027	0.006766	0.8908	0.4387	-0.01551	0.02756
X Variable 1	0.2371	0.01029	23.03	0.000179	0.2043	0.2700

Table 1 represents the regression statistics and the confidence intervals for the calibration curve. As shown above, the y-intercept of the curve is at 0.006027. The lower confidence level is at –0.01551. Subtracting 0.01551 from 0.006027 = -0.009483, proving that zero lies within the confidence interval. Therefore, the y-intercept coefficient is reasonable. WHY DID YOU DO AN ANOVA TEST, AND WHAT INFORMATION FROM THIS STATISTICAL TEST IS RELEVANT?

Table 2. Tabulated Concentrations for Two Unknowns

Unknown #	Avg. Absorbance	[Unknown] ppm	Actual ppm	% Error
1	0.025	80±2	100±10	20.0
2	0.040	142±3	150±15	5.33

Table 2 illustrates the comparison between experimental concentration value and the actual value. The experimental concentration value of unknown 1 lies outside of the range of actual concentration value, deeming this value unacceptable. Experimental concentration value of unknown 2 lies within the range of actual concentration value.

ANALYSIS

The calibration curve data reveals a strong linear relationship ($R^2 = 0.9944$) between absorbance and concentration that can be represented by the equation $y = 0.2371x + 0.006$ (Figure 2). It was determined that the upper limit for the linear working range of the ion being analyzed, Na, was 1.0 ppm.[1] Because this is such a dilute solution, caution must be exercised during dilution and measurement, since even a small error can result in huge percentage uncertainties.

The point (0,0) in the calibration curve data was included because deionized (DI) water should have an absorbance of 0, as the spectrophotometer was zeroed with DI after every measurement (YOU SHOULD MENTION THIS EARLIER, AND COULD POINT OUT THAT YOU WOULD GET A SLIGHTLY DIFFERENT SLOPE AND INTERCEPT IF THE ZERO VALUE WAS NOT INCLUDED IN THE REGRESSION).

[1] Litt, Mitchell. Atomic Absorbtion Spectrophotometer. *Bioengineering Laboratory Manual*. pp. 1-27, 2000

Therefore the y-intercept of the line of best fit (y = 0.006) should not be significantly different from zero. This is confirmed by applying the 95% confidence interval to test significance (see Table 1). The maximum fractional error method for obtaining percentage errors was utilized to find the error for points other than (0,0). This method was chosen to account for all possible instrumentation error. Total instrumentation error amounted to ±2.1%.

Initial spectrophotometry measurements revealed that unknowns 1 and 2 had concentrations of 80 ± 2 ppm and 142 ± 3 ppm, respectively. This represented a tenfold dilution from the standard. YOU SHOULD STATE WHAT THE EXPECTED VALUES WERE, OR POINT OUT THAT THEY ARE LISTED IN TABLE 1. In order to increase accuracy, absorbance values obtained from the highest concentration dilutions in each of the two unknowns were chosen as the basis of experiment. This method resulted in 20% error for unknown 1, and 5.33% error for unknown 2. After obtaining the initial results, a more accurate determination of the concentration of the two unknown solutions could have been determined by re-diluting and re-measuring each solution under the spectrophotometer at a dilution that was closer to the expected value.

In both cases, the experimental results for the unknown solutions were less than the known lab values. Imperfections in the experiment resulted primarily from two factors – systematic error and the extreme dilution of the Na required. Systematic errors could have been introduced in many of the steps of the experiment. One source of error was likely the method of mixing the diluted solutions. When the contents of the volumetric flask were mixed, a cover was placed on top of the opening and the flask was then inverted and shaken. Some Na ions may have adhered to the cover, resulting in a lower concentration when the cover was removed. Also, the solution may not have been mixed enough, resulting in an uneven distribution of Na thus reducing the accuracy of the absorbance value. One other possibility is that a small amount of Na adhered to the pipet tip and was not transferred into the volumetric flask. Any error in this area would have been magnified because of the low concentrations that were used in the experiment. In fact, the higher percentage error in the first unknown solution with respect to the second (a discrepancy of +14.7%) could also be due to the fact that the original concentration of that solution is significantly less than that of the second.

Despite these accuracy errors, the data collected was (SHOULD BE WERE, SINCE DATA ARE PLURAL) precise (NOT REALLY, THE DEVIATIONS WERE >5%). Three trials were conducted per dilution for each sample (YOU SHOULD MENTION THIS EARLIER). For the first unknown, amongst the three trials, the average absorbance was 0.025 ±0.000% and for the second unknown the average absorbance was 0.040 ±0.002%, proving high precision. A suggestion for improving the system and therefore reducing systematic error is to measure the volumes using a mass scale. Based on the fact that 1 ml of water (and also the dilute Na solution) is exactly 1 g, one can obtain an exact measurement of volume based on its mass. Such measurements would be more accurate than measuring the volume based on the 50-ml volumetric flask, since the utilization of the flask requires the volume of the liquid to be estimated based upon where the bottom of the meniscus lies. This improvement would decrease systematic error. YOU HAVE NOT ESTIMATED WHAT THE ERROR IN CONCENTRATION MIGHT BE.

To obtain improved accuracy, it would be ideal to eliminate as much error as possible from the apparatus, methods, and procedures. Since the main apparatus used was the spectrophotometer, eliminating error in its readings will lead to increased accuracy of the data obtained. To do such, one would have to make sure the wavelength setting of the machine is suitable. For example, the proper wavelength of sodium is 589.6nm, but because the band width is narrow at this frequency, it is difficult to get a steady reading. Being that there was a small area under the curve, absorption readings would fluctuate between .050-.150, for any given unknown concentration sample. To correct this problem the wavelength had to be slightly modified to 589.0nm – a similar but less frequent curve – such that more area under the curve is available and the readings would become more stable, fluctuating by only 0.002-0.003 in the absorption readings.

To obtain more accurate results through methods, the types of measuring devices have to be examined. Pipettes have an error of 0.5%. Graduated cylinders have an error of 0.5%, while the volumetric flask has an error of only ±0.08%, and should therefore be used over the graduated cylinders. BUT YOU DIDN'T USE THIS INFORMATION TO ESTIMATE WHAT THE CONCENTRATION ERRORS ARE. As discussed above, the

volumetric flask can be filled according to mass of the solution on a mass balance. This will eliminate poor meniscus readings and parallax error.

Procedurally, it is always ideal to get as many readings as possible in order to validate the precision of the obtained readings.

In conclusion, in this experiment, calibrations curve of atomic absorption versus concentration for Na was constructed and the concentration of two unknowns was determined by correlating their respective absorbance values to the constructed calibration curve. Data collected for the calibration curve showed a linear relationship between absorbance value and concentration. Experimental values of the two unknowns showed that unknown 1 was at a concentration of 80 ± 2 ppm, and unknown 2 was 142 ± 3 ppm. Compared to the actual value, this correlated to percentage errors of (NEGATIVE) 20.0% and (NEGATIVE) 5.33%, respectively. YOU SHOULD POINT BOTH WERE LESS THAN THE ACTUAL VALUES.

The experimental and educational objectives were both met. Group members were introduced to quantitative analysis and analytical techniques as atomic absorption spectrophotometry was conducted in an attempt to determine the concentration of Na.

REFERENCES

Castellan, GW. *Physical Chemistry,* 3rd Edition. Reading, Massachusetts, 1983, Chapter 24.

Litt, Mitchell. Atomic Absorbtion Spectrophotometer. *Bioengineering Laboratory Manual*. pp. 1-27, 2000

APPENDIX

Raw data for Experiment 3. THE ENTRIES ARE AN AVERAGE OF 3 TRIALS AS YOU INDICATED IN THE ANALYSIS SECTION. THE RAW DATA SHOULD INCLUDE ALL OF THE INDIVIDUAL TRIALS IN BOTH TABLES.

Figure 1. Calibration Data (Na)

Sample #	Vol. Na Added (L)	Total Vol. (L)	Dil. Factor	[Na] ppm	Absorbance	Uncertainty (±)
				0	0	0
1	5.0E-05	0.05		1.0	0.237	0.00498
2	4.0E-05	0.05		0.8	0.194	0.00407
3	3.0E-05	0.05		0.6	0.158	0.00332
4	2.0E-05	0.05		0.4	0.105	0.00221
5	1.0E-05	0.05		0.2	0.088	0.00185

THERE ARE NO VALUES IN THE COLUMN FOR THE DILUTION FACTOR.

Figure 2. Absorption Data for Unknowns

Sample #	Vol. Na Added (L)	Total Vol. (L)	[Na] ppm	Absorbance	Avg. Absorbance	[Unknown] ppm	Actual ppm
1a	5.0E-05	0.05	1.0	0.025	0.025	80	100
1b		0.05	0.0	0.025			
1c		0.05	0.0	0.025			
1d		0.05	0.0				
2a	4.0E-05	0.05	0.8	0.041	0.040	142	150
2b		0.05	0.0	0.040			
2c		0.05	0.0	0.038			
2d		0.05	0.0				