Lab 3
Determination of Sodium in Tonic Water by Flame Atomic Absorbance

Introduction to the Lab. Sodium is an important part of the human diet, but given its prevalence in flavor enhancers and preservatives, sodium is often at too high of a level in the human diet. Sodium is implicated in hypertension, a major killer in the US. In a previous lab, you determined the amount of benzoate in tonic water by UV-visible absorbance spectroscopy. According to the label on the bottle, benzoate was introduced in the form of sodium benzoate, which is a preservative. The purpose of this lab is to determine if the amount of sodium in the tonic water corresponds to the amount of benzoate in tonic water, and to compare the amount of sodium to the amount stated on the product’s label. Sodium is routinely analyzed by atomic absorbance spectroscopy (AA or AAS), where the sample is atomized by a flame (FAA) or in a graphite furnace (GFAA). In this lab, you will use flame atomic absorbance spectrometry.

The Matrix. The matrix is everything in your sample aside from the analyte that you wish to determine. As you learned in class, the efficiency of atomization in a flame depends on the matrix because ingredients such as sugar and anything else present affect flame temperature and electron density. In order to be analyzed in the flame AA, your analyte must be atomized and then it must be able to be excited to an upper energy level, and in the process it absorbs light in the spectrometer. If the matrix prevents this from happening in a reproducible way, or if it affects the fraction of your analyte that can be excited, the results of your analysis will lack precision and accuracy.

Standard Addition Method. To account for this, the method of standard additions is typically used. It is a kind of in situ standardization method. The method of standard additions employs the “trick” of spiking the sample with a small volume (or several spikes of small volumes) of concentrated standard, where the small volume avoids changing the composition of the matrix significantly, and the high concentration imparts an additional absorbance comparable to that of the sample. The beauty of the standard addition method as a method of standardization is that you are analyzing your standards in the same matrix as your sample. It is especially useful for “real world” samples (as opposed to idealized chemistry lab samples where your analyte is dissolved in de-ionized water), since one often does not know about the matrix of their sample.

PRE-LAB ASSIGNMENT

Turn this in to your TA at the beginning of your lab period. Print out and keep a second copy to refer to during your discussion with your TA. Be prepared to discuss the issues.

1. Assuming that sodium was added as sodium benzoate, what would be the molar concentration of sodium, based on your results from Lab 2, where you determined the benzoate concentration in tonic water?
2. The following data will help you design your experiment. Using a sodium lamp operating at \( \lambda_{\text{Na}} \) and our atomic absorbance spectrometer, the absorbance of a blank was measured to be 0.008. The absorbance of a \( 1 \times 10^{-4} \) M solution of NaCl was measured to be 0.336. Calculate the molar absorptivity \( \varepsilon \) of sodium in units of M\(^{-1}\) cm\(^{-1}\) at this wavelength.
Assume that the sample length is the length of the flame, or 10 cm. Estimate the absorbance you expect from tonic water.

3. We will provide a standard solution of 0.100 M NaCl and a solution of 20-fold diluted tonic water (the unknown) for your use. The whole class will use these reagents and we will compare all results. Design a range of five volumes of the NaCl standard solution that you will spike into equal aliquots of the unknown. First decide what volume of tonic water unknown to add to each 25-mL volumetric flask. Try to have the third out of five spiked solutions produce approximately double the absorbance of the diluted tonic water. We found that the spectrometer would not register a reliable absorbance much greater than 0.5, so try not to exceed this. You may have to dilute the 0.100 M NaCl stock solution before making your standard additions.

4. Plot the expected calibration curve of absorbance vs. moles Na added. Explain how you will use this curve to calculate the molarity of Na in the diluted tonic water solution.

EXPERIMENTAL

1. Prepare your spiked solutions. As always, use your best quantitative technique in all things you do in this lab. When taking your liquids from the NaCl stock solution and the tonic water unknown, be careful not to contaminate them. Always pour out from the original containers into a clean container that you will use. Never pipette directly from a communal container!!

2. Measure the absorbances of the spiked and unspiked solutions. Use the manual READ button in the software. Check to see how much the baseline signal (the signal with no sample in the beam) drifts by regularly zeroing the instrument prior to your absorbance measurements.

3. Although the software is capable of generating a standard addition plot if you set it up by telling it your concentrations and volumes, you should write down the absorbance values using the manual READ function, and analyze your data in your own spreadsheet.

WRITTEN REPORT

1. Plot your experimental calibration curve, fit it to a line, and report the slope $m$ and intercept $b$ with their standard errors: $m \pm e_m$ and $b \pm e_b$.

2. Calculate the concentration of sodium in tonic water in molarity units, and report the 95% confidence interval.

3. Compare the molarity of sodium determined here with that of benzoate determined previously. Accounting for the confidence intervals in both measurements, state whether they agree within experimental error.

This laboratory was created by Professor Mary J. Wirth, September, 2002. It was revised by Professor Thomas P. Beebe, Jr., September 2003.

We welcome your comments on how to improve the learning experience of this lab. The best time to communicate these comments is when you are working on the lab.