Determination of Sodium in Tonic Water by Flame Atomic Absorbance

Introduction. 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 this lab, you will determine the amount of sodium in a tonic water sample. Although the main ingredient of tonic water is quinine, it also contains sodium benzoate, $NaCO_2C_6H_5$, which acts as an antibacterial agent – 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, if the sodium concentrations are low, in a graphite furnace (GFAA). In this lab, you will use flame atomic absorbance spectrometry. In another laboratory you will determine the amounts of quinine and benzoate in the tonic water sample by UV-visible absorbance spectroscopy. You will also determine the amount of quinine in tonic water by fluorescence spectroscopy.

The Sample Matrix. The sample matrix is everything in your sample other than the analyte that you wish to determine. As you learned in lecture, the efficiency of atomization in a flame depends on the *matrix* because these other ingredients such as sugar and anything else that is present, including the water can affect both the flame temperature and the abundance of free electrons in the flame. In order to give an absorption signal in flame AA, your analyte must first be atomized and then it must be excited by light to an upper energy level. In the process it absorbs light photons from the spectrometer's source. If the matrix prevents this atomization and absorption from happening in a reproducible way, the precision and accuracy of your analysis will be degraded.

Standard Addition Method. To account for components of a sample matrix affecting the size of the analyte signal, 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 with several spikes of small volumes) of concentrated standard, where the small volume avoids changing the composition of the matrix significantly, and the high concentration of analyte imparts an additional absorbance. Ideally, this additional signal should be comparable to half of that produced by the sample alone. The beauty of the standard addition method as a method of standardization is that you are analyzing your standards in almost exactly the same matrix as that of your sample. It is especially useful for "real world" samples (as opposed to idealized chemistry lab samples where your analyte is dissolved in deionized water), since one often does not know full details about the matrix of the sample.

Read more on standard additions in Skoog (6^{th} Ed., pgs 13-17) or Harris (8^{th} Ed., pgs 106-108). Harris has a useful Excel sheet that may be of interest.

Note. If we wanted to determine the amount of benzoate to compare it with the sodium determined here, we would need to use "regular" (not diet) tonic water in these experiments,

because diet soda often has sodium saccharate added, which would preclude the comparison of sodium and benzoate analyses because there would then be two sources of sodium.

PRE-LAB ASSIGNMENT

Write answers to the following questions in your lab notebook. This assignment is NOT always a guideline for the lab you will do; instead, the assignment will help you think about issues that will be important in the laboratory. You can use this assignment to prepare for the laboratory work.

Be prepared to show these to your TA at the beginning of your lab period. Also be prepared to discuss the issues with your teammates and with the TA.

1. What is the expected molarity of sodium in tonic water based on the product's label: 35 mg sodium per "serving" (they report a "serving" as 240 mL)?
2. The following data will help you design your experiment. Using a sodium lamp operating at λ_{Na} and our atomic absorbance spectrometer, the absorbance of a blank was measured to be 0.008. The absorbance of a 2.5×10^{-4} M solution of NaCl was measured to be 0.230. Calculate the molar absorptivity ε of sodium in units of M^{-1} cm^{-1} at this wavelength. Assume that the sample path length is the length of the flame, or 10 cm. Estimate the absorbance you expect from tonic water with the molarity of sodium calculated above.
3. We will provide a standard solution of 0.100 M NaCl and a solution of 40-fold diluted tonic water (the unknown here) for your use. 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. The aim is to increase the Na absorption with each of these additions; try to have the third out of five spiked solutions produce approximately *double* the absorbance of the diluted tonic water. *Note: We find that the atomic absorption spectrometer you are using will* ***not*** *register a reliable absorbance much greater than 0.5, so avoid exceeding this value.* You may have to *quantitatively* dilute the 0.100 M NaCl stock solution before making your standard additions to keep within the range of the instrument. Remember to record any dilutions you make and the glassware that you use to make dilutions. You will need this information for the report.
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, dry container that you will use for pipetting. Never pipette directly from a communal container!***

2. Your TA will show you how to use the instrument. Note that the flame is started with the software, and not by changing valve settings. It is also turned off the same way, so there is no need to change any valve setting for air or for acetylene. Turn the flame off when you are not actively measuring with the spectrometer. 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. Repeat these measurements several times.
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 obtained using the manual READ function, and analyze your data yourself.
4. Sign and date the logbook.

SAFETY AND DISPOSAL

Note: This atomic absorption spectrometer uses acetylene gas to support a 2500K flame. It is very important that you make sure that sufficient acetylene pressure is present (it should be above 80 psi) in the tank and that the hood over the flame area is functioning, as indicated by the vacuum gauge. All valves are pre-set in this experiment for your safety. Do NOT adjust any valves on the acetylene or air inputs. An error in the acetylene-air mixture could damage the instrument or cause an explosion. Any student who tampers with the acetylene settings will receive a safety violation and will be dismissed from the laboratory.

All solutions are to be discarded in the non-organic jug in the hood.

WRITTEN REPORT

1. Plot your experimental standards addition 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. From the results of step 1, calculate the concentration of sodium in tonic water in molarity units. Propagate errors to estimate the 95% confidence interval.
3. Report the concentration of sodium and its 95% confidence interval.