Determination of Sodium in Snack Foods by Flame Atomic Emission Spectrometry

The objective of this laboratory experiment is to determine the amount of sodium in a snack food (potato chips, corn chips, etc...) using flame atomic emission spectrometry. The experiment will also use the CEM Microwave Sample Preparation System to decompose your sample for analysis.

Experimental Procedure:

Sample Preparation: Accurately mass about 30 g of potato chips into a weighing boat, then transfer these to a blender container which has been previously tared to 0.00 g on a top-loader balance. Record the mass of the weighing boat after the transfer of chips.

Add distilled, deionized water to the blender container to give a total mass of chips and water of about 280 g (massed as accurately as the top-loader balance will allow). Blend the samples, starting at low speed, then switching to high speed blending for approx. 3 minutes. Transfer the homogeneous slurry to a clean bottle.

Sample Decomposition: Using a Pasteur pipet, accurately mass enough of the chip slurry to give approximately 0.25 g chip to a clean, tared teflon microwave bomb. Add 5.0 mL of concentrated HNO_3. You should prepare replicate samples for the decomposition. Prepare the bombs, being certain to place a new rupture membrane into each. Also make certain that a control vessel is placed into the microwave so that the pressure of the reaction can be monitored. Heat these samples using the program "potatos" (yes, I know it is spelled correctly, the Dan Quayle school of spelling has some problems with this word). Allow the samples to cool for approximately 10 minutes, slowly vent the pressure from the control vessel, remove from the microwave, and vent the remaining vessels in the fume hood.

Add 2 mL of 30% H_2O_2 to each vessel (be careful to keep the peroxide off of your skin), reseal the vessels and heat them in the microwave using the program "Peroxide". Vent the vessels as described before, then quantitatively transfer to a plastic bottle, recording the final mass of the transferred solution. Use the Na concentration of that listed on the potato chip wrapper to determine the expected concentration of Na (w/w) in your decomposed chip sample.

Analysis of Sodium in the Decomposed Chip: Dilute your decomposed chip samples using mass/mass dilutions to reach a final predicted concentration of approximately 1 part-per-million (μg/g) for the final analysis. Prepare aqueous standard solutions of Na (from 1000 ppm stock standard) of 2, 1.5, 1.0, 0.5, 0.25 and 0 μg/g using a top loading balance and serial dilutions. Remember that the goal is to maintain 3 significant digit accuracy in knowledge of the concentration of these aqueous standards.

The following section should be accompanied with explanations from your instructor or TA prior to use of the instrument. Determination of the sodium concentration is performed using the

Varian SpectrAA 200 with an air acetylene flame. Adjust the slit setting to a 1 nm width, use the 589.0 nm emission wavelength of sodium, integrate for 5 seconds for each sample with 3 repeated integrations. The SpectrAA will prompt you for the correct samples and will prepare a calibration curve which is used to determine the concentration of sodium in your unknown sample. Using these results, compute the total concentration of sodium in the original chip sample in mg Na per serving. Compare this to the value on the original package and draw conclusions from this comparison. Where would your most likely sources of error come from? What things could be done in your analysis to actually improve your confidence in the accuracy of your final result (think about the possible use of a blank, a reference standard material, or "spiked" sample)?

Microwave Programs Employed

I. "Potatos"

Stage	**1**	**2**	**3**
Power	55	55	55
PSI	20	85	175
Time	800	800	800
TAP	500	500	500
FAN	100	100	100

II. "Peroxide"

Stage	**1**
Power	55
PSI	120
Time	800
TAP	500
FAN	100