Determination of Sodium in Snack Foods by Flame Atomic Emission Spectrometry

INTRODUCTION

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.

Instrument Operation Warning:

Before you begin the collecting data, it is extremely important to read and understand the Standard Operating Procedure (SOP) that describes the operation, precautions and rules to follow when using the atomic absorption/emission spectrophotometer. FAILURE TO OBSERVE ANY OF THE PRECAUTIONS CAN AND WILL RESULT IN A VIOLENT EXPLOSION!

REAGENTS AND APPARATUS

- Snack food sample
- 18 MΩ water
- Concentrated nitric acid
- 30% hydrogen peroxide
- Blender
- Microwave decomposition system and vessels
- Atomic emission spectrophotometer

PRE-LAB CALCULATIONS

1. A sodium solution is prepared by weighing 1.24 g of 1000 ppm Na stock solution into a plastic bottle and diluting to a final mass of 23.81 g with high purity water. A portion of this sample weighing 1.08 g was transferred into a second bottle and diluted to a final mass of 48.37 g. What is the sodium concentration in ppm in the final solution?

2. A package of potato chips states that it contains 170 mg sodium per 28 gram serving of chips. A 22.0 gram sample of these chips is blended and diluted with high purity water to a produce a slurry with a final mass of 208.6 grams. What mass of this slurry contains 0.25 grams of potato chips?

PROCEDURE

Instructions for Solution Preparation:

You are to perform all dilutions in this experiment **by mass** on a top-loading balance, rather than by volume. **Do not use pipettes and volumetric glassware.** Use only 18 MΩ highly purified water for these analyses. A carboy will be available for these purposes.

1. **Sample Preparation:** Accurately mass about 15 - 30 g of snack food into a weighing boat and then transfer these to a blender container which has been previously tared to 0.00 g on a top-loading balance. Record the mass of the weighing boat after the transfer of snack food.

   Add distilled, deionized water to the blender container to give a total mass of chips and water of about 10 times the original sample mass (massed as accurately as the top-loading 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.

2. **Sample Decomposition:** Acquire and clean Teflon microwave vessels to prepare triplicate samples for the analysis. Using a Pasteur pipet, accurately mass enough of the slurry to give approximately 0.25 g of the **original snack food** to each clean, tared, Teflon microwave vessel (NOTE: This will require more than 0.25 g of slurry!!). Add 5.0 mL of concentrated HNO_3 and 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 "Snack Foods".

   After heating, allow the vessels to cool and transfer them to a fume hood. In the fume hood, carefully vent the vessels and quantitatively transfer each to clean, pre-weighed plastic bottles, recording the final mass of the transferred solution. Use the Na content listed on the snack food package to determine the expected concentration of Na (w/w) in your decomposed sample.

3. **Analysis of Sodium in the Decomposed Chip:** Dilute your decomposed 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 approximately 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 three significant digits in knowledge of the concentration of these aqueous standards, therefore the minimum mass you should transfer is 1.00 g. Your final masses of solution should be no less than 20 grams and no more than 100 grams. *Have a plan for preparing these solutions before coming to lab or you will waste valuable lab time!*

   In the analysis of these samples, one must also assess the accuracy of the results. In this lab, we will use "spiked samples" to help us draw conclusions about the accuracy of our results. To do so, mass a similar portion of the digested sample as you did to reach a predicted Na concentration of 1 μg/g (as above) and prior to dilution to the target final mass, use a Na standard to add a "spike" of Na that when diluted adds the equivalent of 0.5 μg/g Na to your samples. *Again, having a plan for preparing these solutions before coming to lab will save you valuable lab time!*

   When your solution prep is complete, you will have a total of 12 solutions: 6 standards, 3 unspiked samples and 3 spiked samples. Be sure to calculate the actual concentrations of your standards prior to making your instrumental measurements.

   Determination of the sodium concentration is performed using an atomic emission spectrophotometer with an air acetylene flame. See the **Instrument Operation Warning** above before using the instrument! Tune the instrument to the 589.0 nm emission wavelength of sodium, adjust the slit setting to a 0.2 nm width and integrate the signal for each sample. Measure your standards, samples, and spiked samples. Using these results, compute the total concentration of sodium in the original chip sample in mg Na per serving as well as a percent recovery for the analysis.. Compare this to the value on the original package and draw conclusions from this comparison.

POST-LAB CONSIDERATIONS:

Where are the most likely sources of error in this analysis?

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)?