An Updated Method to Determine Nutrient and Toxic Elements in Foods Using Closed Vessel Microwave Assisted Digestion and Inductively Coupled Plasma Optical Emission Spectrometry

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Introduction

- FDA uses a 30 year old AOAC method to measure nutrient elements in foods
- The method lacks several key features that can help ensure accurate analysis of elements in foods such as internal standardization, a multi-point calibration curve, and closed vessel microwave digestion
- Additionally, the method does not satisfy current FDA validation requirements
- This presentation describes the optimization of a new method that measures both macro and micro-nutrient concentrations in all food matrices
- Sample preparation is harmonized with EAM 4.7, the current toxic/trace element analysis method
- Method development and optimization for sodium is used as an example of the process to optimize ICP-OES parameters for the analysis of 23 elements in all foods

Method Summary

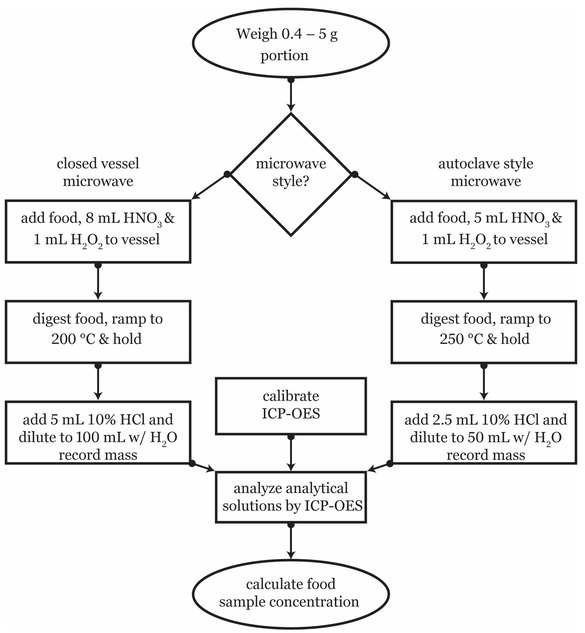

Figure 1. Summary of the working method for foods analysis by ICP-OES. Outline modeled after EAM 4.7

ICP-OES Optimization

- Case Study: Sodium 590 nm line
  - Spectral interferences?
  - Background correction?
  - Matrix effects and ionization buffer?
  - Linear dynamic range?
  - Axial or radial plasma view?
- Analytical results from the analysis of reference materials are used to determine optimal instrumental parameters

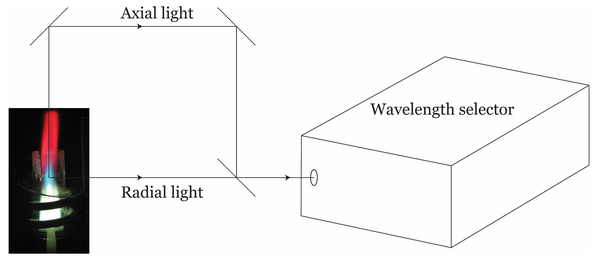

Figure 2. Optical arrangement for radial or axial viewing of the plasma

- Plasma viewing
  - Axial viewing is a more sensitive approach
  - Radial viewing is typically a more precise form of measurement
  - Radial viewing has a larger linear dynamic range
  - Axial viewing requires cesium as an ionization buffer to extend the linear dynamic range
  - Matrix effects are typically less severe for radial viewing

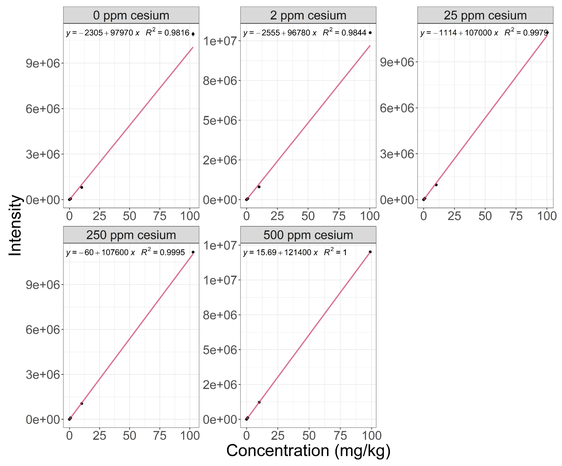

Figure 3. Sodium 590 nm calibration curves at varying levels of ionization buffer. Plasma is viewed axially

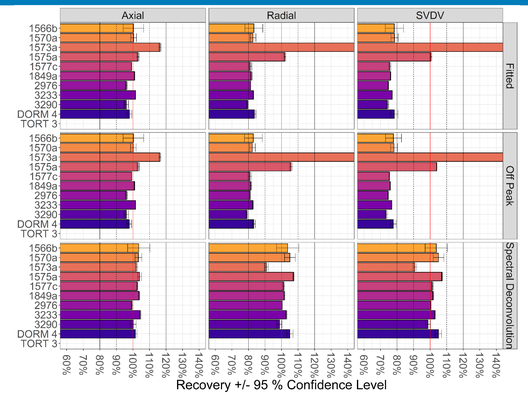

Figure 4. Analytical recoveries of sodium in several reference materials. The 590 nm line was considered in addition to the evaluation of several viewing modes and background and interference correction techniques

- Fitted background correction resolves background and large, distant interfering peaks by applying peak-shaping functions to the analyte peak
- Off peak background correction resolves background by subtracting intensity from a flat background point adjacent to the analyte peak
- Spectral deconvolution resolves background and spectral interference by remembering the spectra of interfering species with emission near the analyte peak

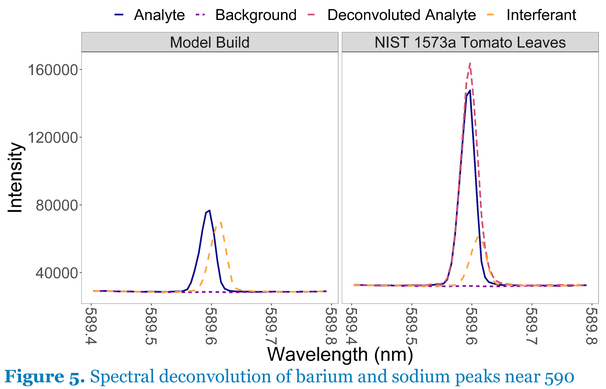

Figure 5. Spectral deconvolution of barium and sodium peaks near 590 nm

- Spectral interference at 590 nm
  - Sodium and barium wavelengths are separated by 20 pm and are unresolved by most OES systems
  - Foods with barium will cause false positives for sodium at 590 nm if not accounted for
  - Spectral deconvolution is needed to resolve background emission and overlapping peaks
  - Axial viewing and spectral deconvolution is suggested for sodium analysis using the 590 nm line

Results and Discussion

- The same optimization process, considering spectral interference, matrix effects, and background correction, is currently being applied to the other 22 elements

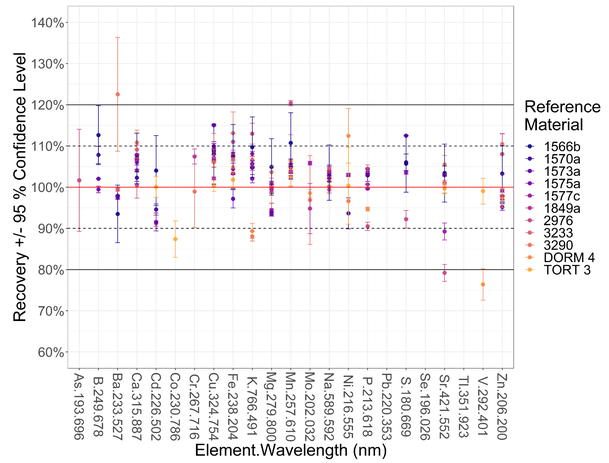

Figure 6. Summary of the analysis of 11 CRMs by the ICP-OES method in development. Duplicate portions of CRMs were analyzed

- Positive bias may indicate unresolved spectral interference or matrix effect
- Current work involves evaluating the utility of interelement correction and spectral deconvolution for wavelengths with potential spectral interference

Conclusion

- A method is in development to meet current U.S. Food and Drug Administration needs for accurate nutrient element analysis of foods
- Key ICP-OES parameters to improve accuracy and robustness include plasma viewing orientation, background and spectral interference removal techniques, and the amount of ionization buffer in the plasma
- Accurate results are shown for several CRMs for most elements considered
- Immediate plans include additional analysis of CRMs with elevated levels of As
- An FDA level two single lab validation will be carried out to evaluate the method's accuracy across the AOAC food triangle