Method Optimization for the Analysis of Food, Dietary Supplements and Cosmetics by LA-ICP-MS

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Abstract
In this study, we present the development and optimization of an analytical method based on Laser Ablation – Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS) that can be applied to food, dietary supplements, and cosmetic products. In this method, samples were ground and mixed with a cellulose powder containing internal standards and multivitamin standard reference material (SRM); the resulting powder was pressed into pellets. The samples were ablated using a UV laser followed by ICP-MS analysis of the removed solid. Different laser and ICP-MS parameters were closely monitored to produce the best sensitivity while reducing the variability between replicate measurements. Samples of unknown multivitamins and reference materials were analyzed by LA-ICP-MS using the optimized parameters and the results were compared to reported values. An experiment was conducted to determine the effect of different focal points in the ablation of pellets.

Introduction
• Monitoring nutritional and toxic elements present in food and cosmetics is part of the Food and Drug Administration’s mission to protect and promote public health.
• We present the development and optimization of an analytical method based on LA-ICP-MS that can be applied to food, dietary supplements, and cosmetic products.
• This technique offers quick and automated sample analysis without the need for corrosive acids and extensive sample preparation.
• This method is especially useful for products that are resistant to nitric acids, such as dietary supplements.
• Integration of a carousel autosampler allows for automated and unattended analysis, resulting in high sample throughput.

Materials and Methods
• Cellulose multielement powders were prepared by mixing ICP-MS standards followed by drying and homogenization.
• The standard powders were further characterized by solution (following a modified EAM 4.7 method) and laser ablation ICP-MS.
• The calibration standards were prepared by mixing the multielement reference material NIST 3280 and 2 g of internal standard for a total of 2 g of internal standard and pressed into pellets (Figure 1).
• The pellets were ablated and analyzed using an excimer-based laser ablation system (IONBROX, Elemental Scientific Lasers LLC) coupled to an ICP-MS instrument (Icap Q, Thermo Fisher Scientific).

Results and Discussion
• Table 1 summarizes the optimized parameters for the analysis of multivitamins by LA-ICP-MS.
• Figure 3 (left) shows the intensity counts obtained after ablation at different energy outputs, and Figure 3 (right) shows the relative percent difference between cellulose and NIST 3280 for rhodium.
• The optimum energy output selected was 0.5 J/cm² (5% laser power) which resulted in only minor differences in ablation between the two materials, without significantly sacrificing sensitivity.
• Figure 4 shows the LA results for the standard reference materials and three unknown multivitamins for a load in comparison to the certified and solution values.
• Figure 5 shows the relative percent difference (RPD) of the intensities after raising and lowering the stage from -100 to 100 µm away with respect to the center focal point.
• The dissimilarities in ablation yield due to differences in focal points were successfully corrected after normalizing to the internal standard, resulting in RPD of less than 10%.

Conclusion
• We present the development and optimization of a LA-ICP-MS method that offers quick and automated sample analysis without the need for corrosive acids and extensive sample preparation.
• The optimum energy output selected was 0.5 J/cm².
• Reference materials and unknown multivitamins were analyzed using the optimized method.
• The laser ablation ICP-MS recoveries were compared to the certified and/or solution ICP-MS values.
• Dissimilarities in ablation yield due to differences in focal points were successfully corrected after normalization to the internal standard, resulting in RPD of less than 10%.

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