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 acid digestion and require the use of hydrofluoric and/or perchloric 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 cellulose powder at increasing concentrations with 1 g of multivitamin reference material NIST 3280 and 2 g of internal standard for a total mass of 4 g
- The unknown multivitamins were ground, mixed with 2 g of internal standard, and pressed into pellets (Figure 1)
- The pellets were ablated and analyzed using an excimer-based laser ablation system (NWR193, Elemental Scientific Lasers LLC) coupled to an ICP-MS instrument (iCap Q, Thermo Fisher Scientific)

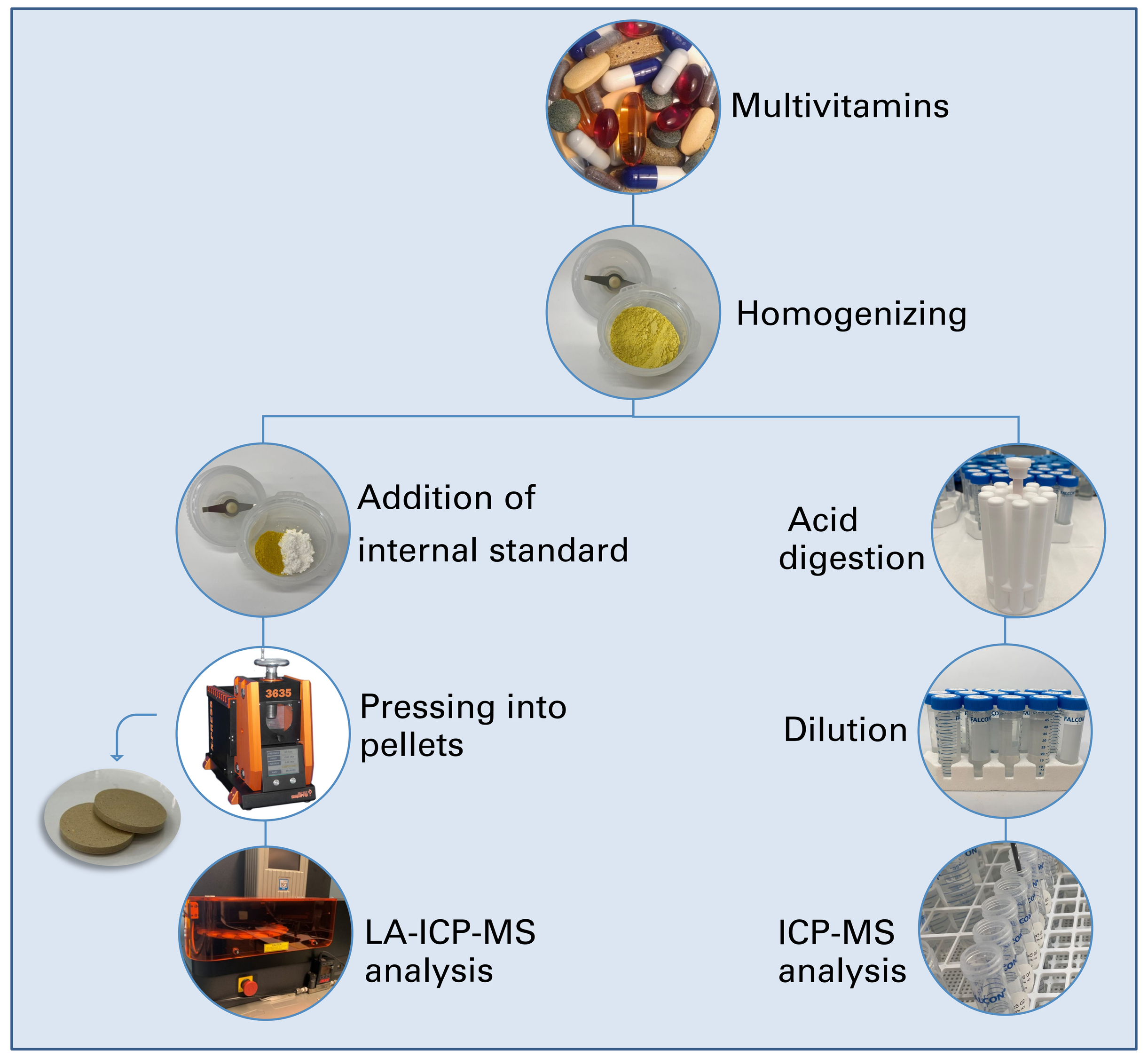

Figure 1. Diagram of the sample preparation sequence for laser and solution ICP-MS.

Figure 1 shows a diagram for the sample preparation sequence for laser ablation and solution ICP-MS analyses. All samples were analyzed by two different solution ICP-MS instruments as confirmatory techniques. Figure 2 shows the schematic representation of the laser ablation and ICP-MS analysis, highlighting laser the ablation cell.

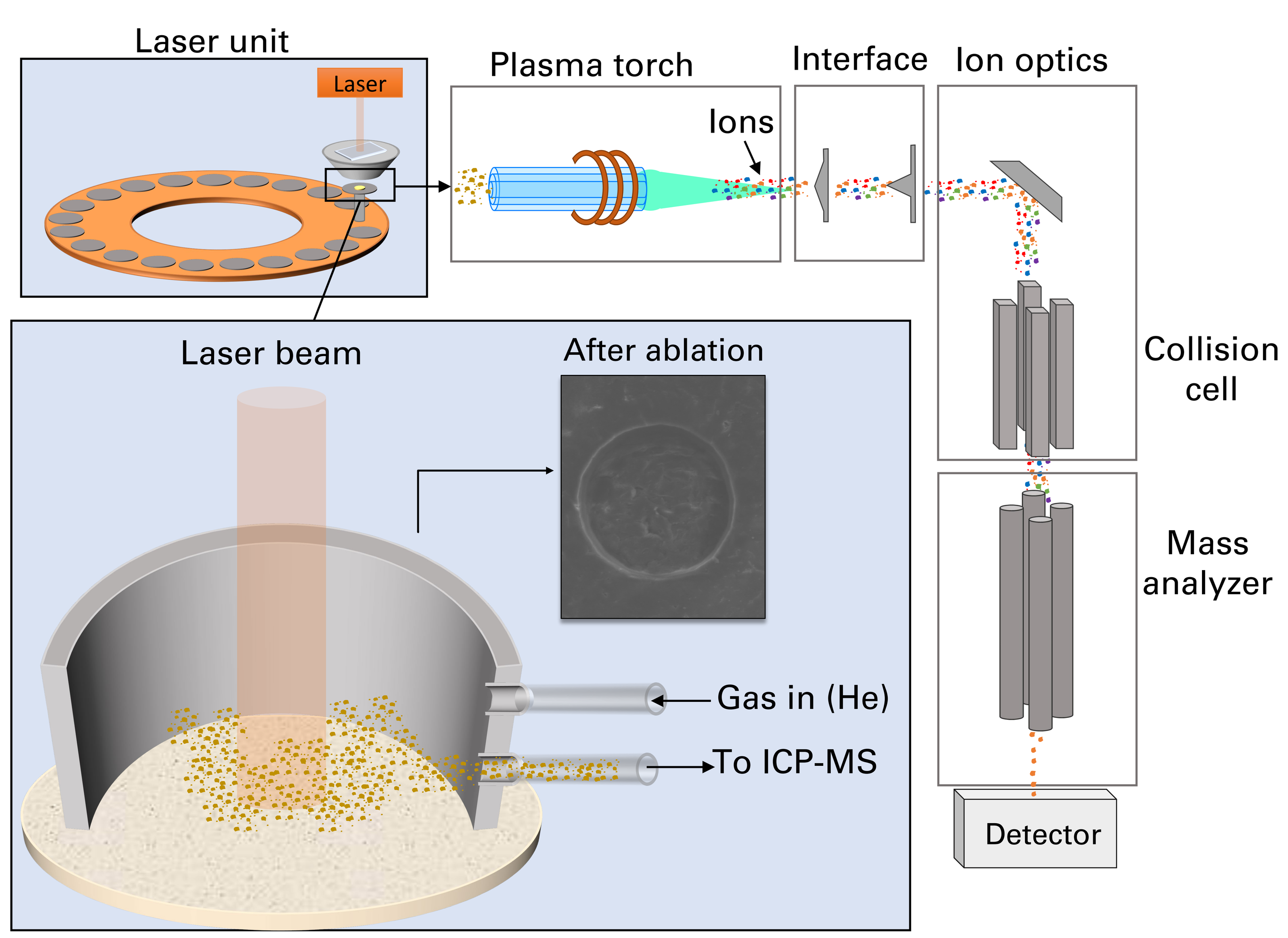

Figure 2. Schematic of the LA-ICP-MS instrumentation.

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 ablating 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 lead 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%

Table 1. Optimized laser and ICP-MS parameters.

Instrumental Parameter	Optimized Value
Fluence	0.50 J/cm²
Frequency	30 Hz
Scan speed	50 µm/s
Spot size	150 µm
Carrier gas flow, He	800 mL/min
Makeup gas flow, Ar	400 mL/min
Additional gas, N_2	5 mL/min
RF power	1550 W
Dwell time	10 – 50 ms
KED mode	He, 4.8 mL/min
Analytes monitored	B, C, Na, Mg, P, S, K, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Cd, Cs, Ba, Hg, Pb, Bi, Th, U

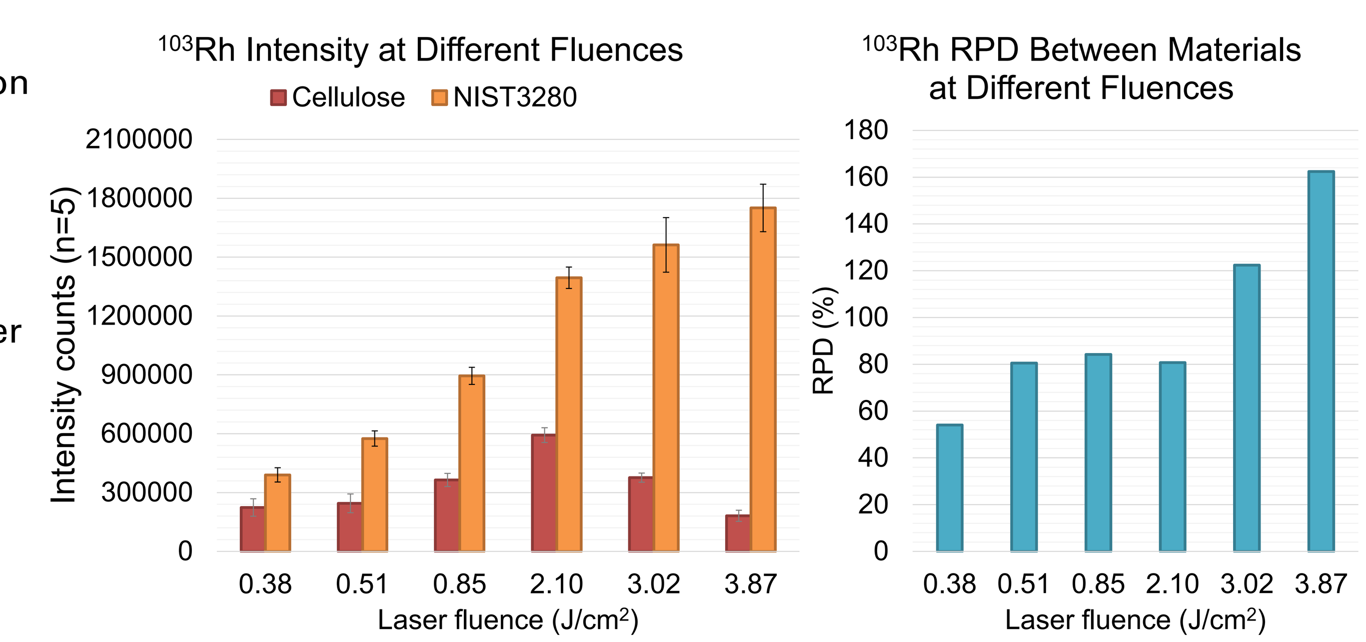

Figure 3. Left: Intensity counts for cellulose and NIST 3280 pellets at different fluences. Right: Relative Percent Difference (RPD) between the intensities of cellulose and NIST3280 at each fluence.

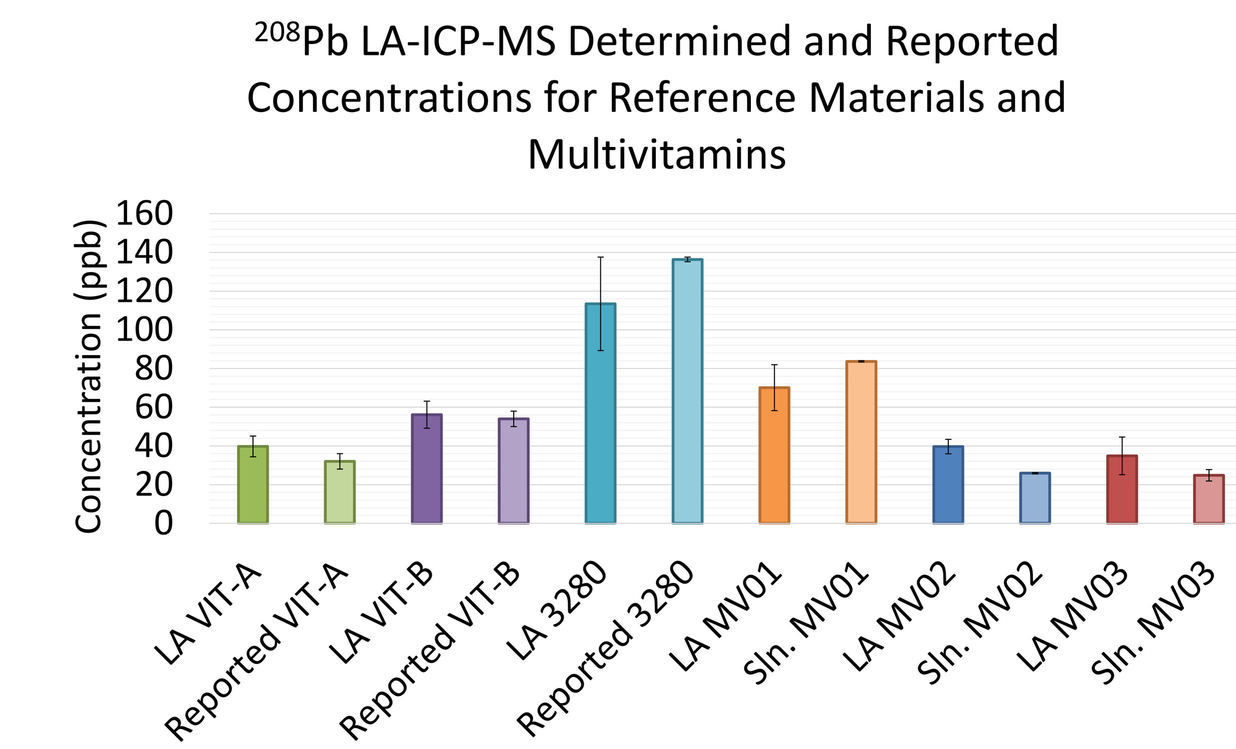

Figure 4. LA-ICP-MS reported concentrations (ppb) (for the SRM) and those determined from solution ICP-MS (for the unknown multivitamin samples).

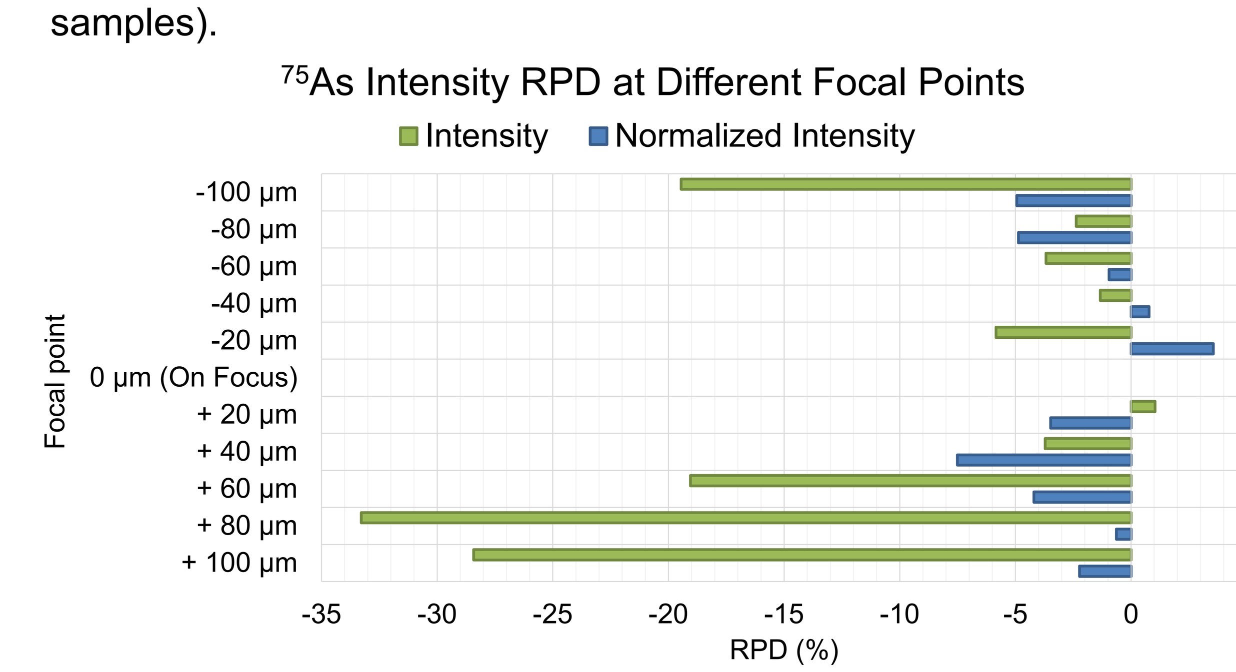

Figure 5. Relative Percent Difference (RPD) at several focal points before and after signal normalization for arsenic.

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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