

ICP-OES Analysis of Nutrient Elements in Dietary Supplements for Accurate Product Labeling

Analyzing complex and varied samples by Agilent 5900 SVDV ICP-OES following FDA EAM 4.4

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Figure 1. A selection of dietary supplements.

Rising popularity of dietary supplements

Consumer demand for dietary supplements is increasing year on year, driving sales and product investment. Dietary supplements typically contain vitamins, minerals, amino acids, herbs, botanicals, concentrates, extracts, or metabolites in the form of tablets, capsules, gels, liquids, or powders.

The US Food and Drug Administration (FDA) Food, Drug and Cosmetic Act and Dietary Supplement Health and Education Act of 1994 include directives that are relevant to dietary supplements. Manufacturers and distributors are prohibited from selling adulterated or misbranded products and are responsible for the appropriate labeling of nutrients, accuracy of health-benefit claims, and product safety.

Smart tools ensure consistent and compliant results

The Agilent 5900 SVDV ICP-OES coupled with the Agilent SPS 4 Autosampler was used for the high throughput, time efficient analysis of dietary supplements. The 5900 was fitted with the default sample introduction system and 1.8 mm Easy-fit one-piece vertical torch, ensuring a simple and repeatable setup. [Early maintenance feedback](#) tracked instrument performance and alerted the user of any maintenance actions during the analysis. Other smart tools within the [Agilent ICP Expert](#) Pro software including [IntelliQuant Screening](#) and [Intelligent Rinse](#) assisted with method development and method optimization times.

The 23 elements specified in the US FDA Elemental Analysis Manual 4.4 (plus sulfur) were included in this study: Al, As, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, Se, Sr, Tl, V, Zn, and S. The EAM 4.4 method assists the analyst in complying with analytical requirements for labeling of elemental nutrients and micronutrients in dietary supplements. It also provides guidance on identifying the elemental composition of samples.

Automatic optimization of rinse times

There can be a wide range of elements and concentrations across different types of dietary supplements, making it difficult to know how long the rinse-cycle should be between samples. With Intelligent Rinse, the software automatically controls rinse times based on user-defined thresholds, saving time and resources while maintaining instrument performance.

In the run shown in Figure 2, 275 solutions were analyzed by the 5900 ICP-OES. The rinse time would be 25% faster using Intelligent Rinse (orange line) compared to standard rinse conditions (blue line), corresponding to a time difference of over 60 minutes. Intelligent Rinse also saves on rinsing solution, argon usage, and power.

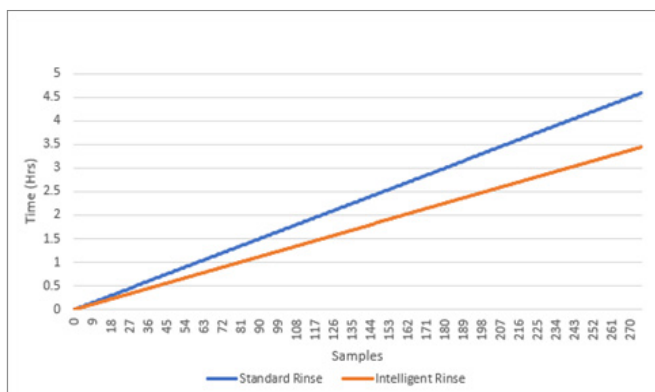


Figure 2. Rinse time for 275 solutions with and without Intelligent Rinse.

Which elements are in my samples?

The supplement industry is constantly developing and releasing new products to meet consumer demand. It is therefore difficult to predict an estimated elemental content of samples due to the wide variation in matrices, presenting a challenge for the analyst. The IntelliQuant Screening software feature can screen samples in a few seconds and deliver semiquantitative data on elemental concentrations before calibration standards are prepared. As the software scans for over 70 elements, it can identify contaminants introduced during production or QA/QC testing that might otherwise go undetected. IntelliQuant Screening data can be presented visually, including as a pie chart (Figure 3), providing a useful overview of the elemental composition of a sample.

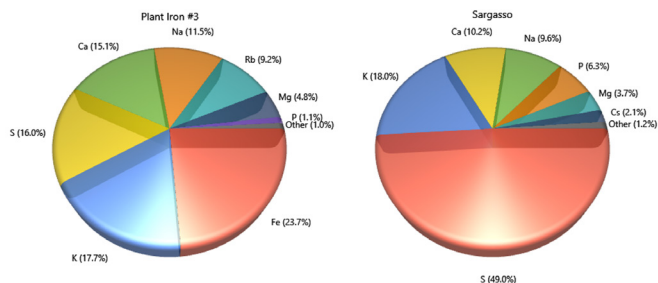


Figure 3. Pie charts of IntelliQuant data for plant iron (left) and sargasso (right).

How do I select the best wavelength?

Choosing the best wavelength for a method greatly affects the success of an ICP-OES analysis. IntelliQuant Screening helps identify analyte lines that are best suited to the concentration range of a sample. The IntelliQuant algorithm displays the best wavelengths for each sample using a star-ranking system. The feature also provides insight into alternative lines by identifying potential interferences on a particular wavelength, as shown in Figure 4.

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background	
Na	✓		589.592	*****	4.99420	1631873.5	85960.7	
			588.995	*	5.11540	3117505.7	123742.2	
			568.821	***				75777.7
			330.237	**				4047.7
			615.423	*				274161.7
P	○		213.618	*****			1875.4	

Analyte: Na(588.995)
 Confidence: very weak
 Interference: Ar(588.886)
 Confidence: very strong

Figure 4. IntelliQuant star-ranking feature helps with wavelength selection.

Conclusion

The Agilent 5900 SVDV ICP-OES provides formulators, manufacturers, and distributors of dietary supplements with a routine and accurate method that meets current testing requirements with added flexibility if requirements change.

See the application note for more information: [5994-5874EN](https://www.agilent.com/chem/5994-5874EN)

www.agilent.com/chem/5900icp-oes

DE60360666

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 Printed in the USA, August 23, 2023
 5994-6211EN

