

Elemental Analysis of Functional Foods by ICP-OES

Using the Agilent 5900 SVDV ICP-OES for quick and easy US FDA method EAM 4.4 compliant analysis

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Figure 1. An elderberry juice functional food, promoted for its immune support properties.

Importance of functional food testing

Functional foods, which are foods that supplement the diet to provide health benefits that extend beyond basic nutrition, have seen a marked rise in popularity over recent years. To maintain accordance with nutritional labeling requirements, such as those outlined by the US Food and Drug Administration (FDA), food testing labs need to keep up with market trends. Consumers expect that the foods and supplements they buy are free from adulteration, chemical residues, and contaminants.

The FDA Elemental Analysis Manual (EAM) 4.4 is an example of a method that outlines how to analyze elements in food by ICP-OES for regulatory purposes (1). The EAM 4.4 method was adapted in this study for the analysis of elemental nutrients and micronutrients in elderberry-based functional foods (Figure 1).

Determine all elements in a single measurement

The Agilent 5900 Synchronous Vertical Dual View (SVDV) ICP-OES is designed for high-throughput laboratories that require accurate measurements in the fastest possible time. The 5900 has a unique dichroic spectral combiner (DSC) that enables both axial and radial viewing in a single reading, halving analysis times when compared to other ICP-OES instruments. This feature is ideally suited to food samples, which typically contain elements such as Na and K at elevated levels and other elements at trace levels. Traditionally, major and trace elements would need to be separated into sequential axial and radial runs, but with SVDV they can be analyzed in a single measurement. When used with an internal standard, SVDV mode helps alleviate the effects of easily ionized element (EIE) interferences.

Method development made simple

When presented with an unknown sample, it is useful to perform a semiquantitative scan, as the data can help with wavelength selection and calibration range. IntelliQuant Screening is a routine within the Agilent ICP Expert software that can semiquantify over 70 elements in a sample in a few seconds. The scan data provides the analyst with valuable knowledge about the samples that saves method development time. The IntelliQuant semiquantitative data can be viewed with several displays, including a periodic table heat map (Figure 2). The heat map provides an at-a-glance overview of the sample content, without needing to prepare a full quantitative worksheet for every element.

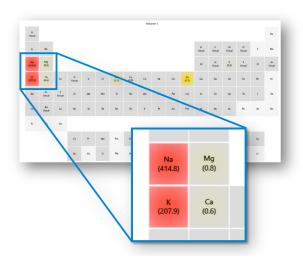


Figure 2. IntelliQuant Screening elemental heat map, showing elements present and their semiquantitative concentration (ppm).

Automatic background correction

The ICP Expert software also contains Fitted Background Correction (FBC), which automatically corrects for both simple and complex background structures. FBC removes the need to spend time manually correcting background peaks using other traditional techniques such as off-peak background correction.

When dealing with unknown matrix types or variable background structures, FBC is highly suitable, as background correction is applied automatically, requiring no extra preparation. Figure 3 shows an example of FBC for Ca 315.887 in a modified elderberry syrup.

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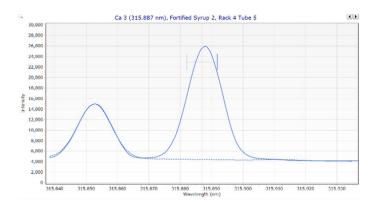


Figure 3. Automatic background correction using FBC for Ca 315.887.

EAM 4.4 compliant analysis

To validate the method per EAM 4.4, a standard reference material and spike recovery tests were performed. All recoveries were all within $100\pm10\%$, well below the stipulated limit of 100 ± 20 (2). Three elderberry-based functional foods were diluted 1:100 and analyzed with the 5900 SVDV ICP-OES. The quantitative limits of detection (LOD) and quantification (LOQ) results in Table 1 were below the nominal limits outlined in EAM 4.4. The data shows the suitability of the 5900 method for the analysis, especially for nutritional labeling of elemental minerals in foods.

Table 1. LODs and LOQs for all elements, dilution factor applied. Units: mg/kg.

Element and Wavelength (nm)	Quantitative Results		EAM 4.4 Nominal Limits	
	LOD	LOQ	LOD	LOQ
Ca 315.887	0.107	0.357	8	30
Cu 324.754	0.0490	0.163	0.1	0.3
Fe 259.940	0.0295	0.0984	0.2	0.3
K 766.491	7.34	24.5	20	40
Mg 285.213	0.0164	0.0546	2	6
Mn 257.610	0.00684	0.0228	0.2	0.4
Na 589.592	0.893	2.98	2	5
P 178.222	1.70	5.67	2	6
Zn 213.857	0.0216	0.0721	0.3	0.8

References

- 1. Mindak, W. R., Dolan S. P., Elemental Analysis Manual, 4.4, Ver 1.1, FDA, Rockville, MD, 2010
- Determination of Elemental Nutrients and Micronutrients in Functional Foods by ICP-OES, Agilent publication <u>5994-</u> 5356EN

