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Application News

No. J124

Inductively Coupled Plasma Atomic Emission Spectrometry

Simultaneous Analysis of Nutrients (Minerals) in Food with Nutrient Function Claims Using ICPE-9820

Introduction

Various Food with Nutrient Function Claims (FNFC) are commercially available for people who take health foods to promote their health or as dietary or supplementary intake of nutrients. FNFCs are a type of Food with Health Claims (FHC) and are allowed to advertise their functionality which is useful in maintaining or promoting health.

Table 1 lists the standards [1] for nutrients (minerals) in FNFCs. Of mineral nutrients, calcium (Ca), potassium (K), iron (Fe), magnesium (Mg), zinc (Zn), and copper (Cu) are allowed indication as a specific health benefit.

Annex Table 9 of the Food Labeling Standards [2] describes inductively coupled plasma atomic emission spectrometry (ICP-AES) as one of the methods for measuring such minerals. In addition, detailed analysis methods for each nutrient are described in an attachment to the "Concerning Food Labeling Standards". [3]

In this research, we conducted simultaneous analysis of nutrients (minerals) within commercially available FNFCs using a Shimadzu ICPE-9820 Simultaneous ICP Atomic Emission Spectrometer. The ICPE-9820 is capable of automatically switching between axial view (AX), which allows high-sensitivity analysis, and radial view (RD), which allows measurement up to high concentrations. Analysis efficiency can be greatly improved by simultaneously analyzing nutrients with different concentrations utilizing both axial and radial view measurements.

C. Kiriyama

Table 1 Upper and Lower Limit Standards for Nutrient (Minerals) Content in the Recommended Daily Intake of FNFCs

Unit: mg

Nutrient	Lower Limit	Upper Limit
Zinc (Zn)	2.64	15
Potassium (K) *1	840	2800
Calcium (Ca)	204	600
Iron (Fe)	2.04	10
Copper (Cu)	0.27	6.0
Magnesium (Mg)	96	300

*1 Potassium is excluded with regard to processed foods in tablet or capsule form.

Sample

Commercially available FNFCs (four products)

Sample A: Calcium, magnesium, zinc
Sample B: Multiminerals
Sample C: Multivitamins and minerals
Sample D: Calcium, magnesium, zinc, vitamin D

Sample Preparation Referring to Analysis Methods of Nutrients [3]

A sample of 1 g was weighed out and incinerated using an electric furnace (500 °C). The ashes were then added 3 mL of 10 % hydrochloric acid and dried on a hot plate. After adding 5 mL of 10 % hydrochloric acid, the sample was covered with a watch glass and heated for 30 min. The sample was then filtered with filter paper, measured up to a volume of 50 mL, and diluted by a factor of 50 to be used as the measurement solution. In order to verify the validity of the measurement method, a spike sample analysis solution was prepared by adding a standard solution of a certain concentration to a digestion solution of sample C.

Instrument and Analytical Conditions

A Shimadzu ICPE-9820 Simultaneous ICP Atomic Emission Spectrometer was used for measurement. Table 2 shows the analytical conditions.

Table 2 Analytical Conditions

Instrument	: ICPE-9820
High-frequency output	: 1.2 kW
Plasma gas flow rate	: 10 L/min
Auxiliary gas flow rate	: 0.6 L/min
Carrier gas flow rate	: 0.7 L/min
Sample introduction	: Nebulizer, 10UES
Chamber	: Cyclone chamber, HE
Plasma torch	: Mini-torch
Observation	: Axial (AX) / Radial (RD)

Analysis

Seven elements were selected for measurement from the FNFCs: Ca, Mg, Zn, and Cu which have standard values, and manganese (Mn) and sodium (Na) which have values indicated on the product package.

The calibration standards were created using commercially available single element standards, and quantitative analysis of the seven elements in the measurement solution and the spike sample analysis solution was performed using the calibration curve method.

Analytical Results

Table 3 shows the quantitative analytical results. The values in the table are indicated converted to an amount equivalent to the recommended daily intake of the nutrient indicated on the respective product. We can see that the obtained analysis values are nearly equivalent to the nutrient information indicated on the products. In addition, the recovery rate for sample C was favorable at 97 % to 104 %, indicating the validity of the measurement method.

Fig. 1 and 2 show spectral profiles and calibration curves. A calibration curve with favorable linearity can be obtained for Ca, which is in the high-concentration range (up to 200 mg/L), by radial view measurement. Furthermore, radial view measurement of Na obtained favorable analysis results (recovery rate: 97 %) because there was no influence from the ionization interference from Ca.

Conclusion

ICPE-9820 enables simultaneous analysis of nutrients (minerals) with widely varying concentrations in FNFCs, thereby improving analysis efficiency.

<References>

(1) Annex Table 11 of Food Labeling Standards (Cabinet Office Ordinance of Mar. 20, 2015)
(2) Annex Table 9 of Food Labeling Standards (Cabinet Office Ordinance of Mar. 20, 2015)
(3) Concerning Food Labeling Standards Attachment - Analysis Methods of Nutrients (CAA/FLD Notification No. 139 of Mar. 30, 2015 issued by the Deputy Secretary General of the CAA)

Table 3 Quantitation Results of Elements in FNFCs (mg/recommended daily intake)

Element	Wavelength	Observation	Sample A		Sample B		Sample C		Sample D	
			Analysis value	Indicated nutrient content	Analysis value	Indicated nutrient content	Analysis value	Indicated nutrient content	Analysis value	Indicated nutrient content
	nm		mg	mg	mg	mg	mg	mg	mg	mg
Ca	183.801	RD	163.8	166.6	245	250	107	100	508	500
Mg	383.826	RD	87.1	83.3	125	125	51.8	50	252	250
Na	589.592	RD	1.52	0 to 5	0.32	0 to 2	1.33	1 to 2	6.95	6.12
Zn	213.856	AX	2.31	2.33	5.73	6	2.50	2.34	6.99	7.0
Fe	238.204	AX	-	-	4.30	4	3.30	2.5	-	-
Cu	324.754	AX	-	-	0.62	0.6	0.22	0.2	-	-
Mn	257.610	AX	-	-	-	-	1.24	1.17	-	-

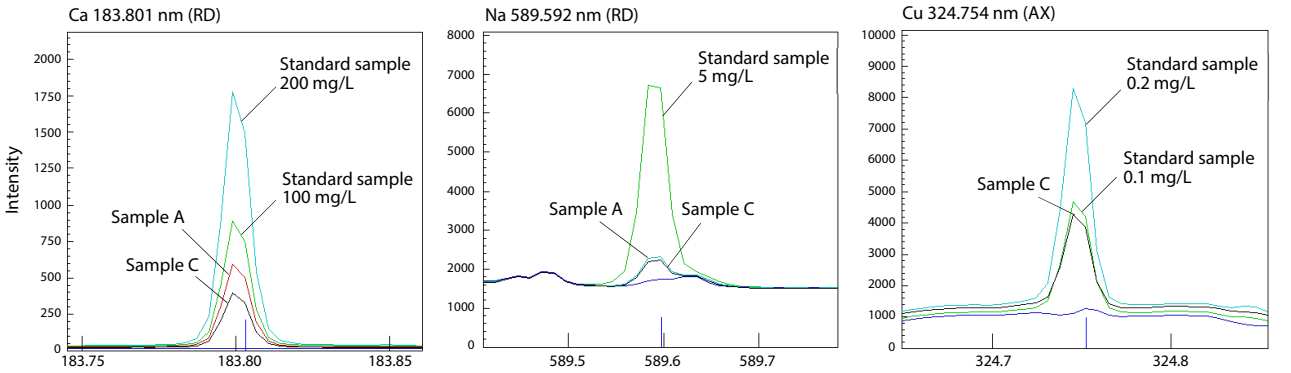

Fig. 1 Spectral Profiles of Ca, Na, and Cu

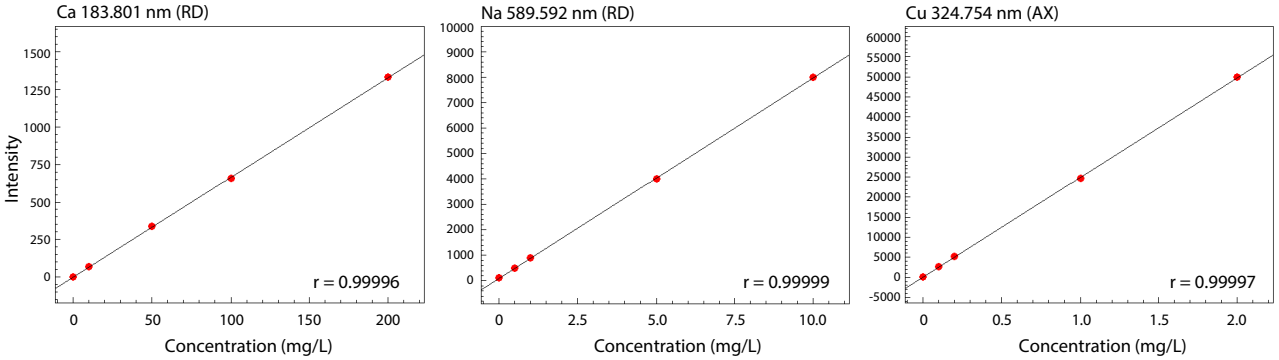

Fig. 2 Calibration Curves for Ca, Na, and Cu

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