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Robust single method determination of major and trace elements in foodstuffs using the Thermo Scientific iCAP 7400 ICP-OES Duo

Authors

Nora Bartsch,
Application Specialist,
Thermo Fisher Scientific,
Bremen, Germany

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Goal

This application note demonstrates the ability of the Thermo Scientific iCAP 7000 Plus Series ICP-OES to determine trace elements and major components in foodstuff to comply with regulations.

Introduction

During the last decades, food protection from potentially hazardous contaminants has become a major topic of public interest. The organization charged with the development of the Codex standards (worldwide food and consumer regulations) is the Codex Alimentarius Commission, which is an intergovernmental body jointly sponsored by the Food and Agriculture Organization and the World Health Organization. As well as the standard regulatory testing, it is necessary to account for contaminants, which may enter the food chain via many means, such as industrial pollution or environmental contamination, i.e. polluted rainfall on crops. Once toxic elements are in the food chain, they can pose significant health risks. With this in mind and the increasing number of micronutrients requiring determination, it is critical that the method of testing is a rigorous and reliable one.

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Instrumentation

The Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES employs a high-resolution echelle spectrometer with a charge injection device (CID) detector. Advancements in CID technology allow this detector to feature higher sensitivity and lower noise than any of its predecessors. An iCAP 7400 Duo instrument was chosen for this analysis as this enables maximum sensitivity using axial view whilst maintaining excellent matrix tolerance in radial viewing mode. The instrument parameters used are listed in Table 1.

Table 1. Instrument parameters.

Parameter	Setting
Pump Tubing (Standard)	Sample Tygon® orange/white Drain Tygon® white/white
Spraychamber	Glass cyclonic
Nebulizer	Glass concentric
Center Tube	2.0 mm
Pump Speed	50 rpm
Nebulizer Gas Flow	0.6 L·min^{-1}
Auxiliary Gas Flow	0.5 L·min^{-1}
Coolant Gas Flow	12 L·min^{-1}
RF Power	1150 W
Exposure Time	UV 15 s, Vis 5 s

Method development and analysis

Initially, more than one wavelength was selected for each element (using multiple views axial/radial). The subarrays for each wavelength were then examined and the most appropriate wavelength for the application was chosen based on the presence of inferences, calibration curve, readback of standards, QCs, and CRMs and the required linearity for the element (Table 2). The subarray plots for each element can be easily manipulated by the analyst, allowing the optimum peak integration and background correction points to be selected. The Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software was used for data acquisition and provides easy options for post-analysis data manipulation. Qtegra ISDS Software has intergraded Quality Control (QC) checks as well as a Flags and Limits function which automatically control the specifications of the method and flag samples accordingly.

Table 2. Element, wavelength and plasma view used.

Element	Wavelength (nm)	View
Ca	317.933	Radial
Cu	327.396	Axial
Fe	274.932	Radial
Mg	285.213	Radial
Mn	257.61	Axial
Ni	231.604	Axial
P	178.284	Axial
Zn	206.2	Axial

Sample preparation

Samples were prepared using certified reference material (CRM) standards. A 0.5 g aliquot of the Total Diet (ARC182) was digested in 5 ml concentrated HNO_3 and 1 ml concentrated HCl. 0.5 g aliquots of the Wheat Flour (NBS1567) and Bovine Liver (NBS1577a) were digested in 9 ml HNO3 using a standard food method program in a high pressure microwave system. The final digests were made up to 50 ml with deionized water before analysis.

Standard preparation

High purity standards (1000 mg·kg^{-1} single element standards) were used to prepare the calibration standards for this method. They were then acid matched to the samples. Table 3 indicates the concentration of each of the standards, selected to cover the linear range of the samples.

Table 3. Standard concentrations.

Element	Concentration in mg·kg^{-1}
Cu, Mn	0.1
Ni	0.5
Cu, Fe, Mn, Ni, P, Zn, Ca, Mg	1
Zn	5
Cu, Fe, Ca, Mg, P	10
Ca, P	50

Results

The instrument was calibrated using a blank and at least two standards for each element. After inspection a linear fit was applied to all elements. The calibration standards and samples were analyzed in a single sequence with a dilute acid rinse (0.5% HNO_3) between samples. The sample data was measured by interpolation and results are shown in Table 4. Suitable dilutions were made to over-range elements to ensure they fell within the calibration range. Method detection limits (MDLs) were also established by analyzing the acid matched calibration blank using a 10 replicate analysis and multiplying the standard deviation of the analysis by 3. This was repeated three times and the average values for detection limits were calculated (Table 4).

Table 4. Results of measurement, CRM and MDLs in mg·kg^{-1} and calculated recoveries in %.

Element	NBS1577a			NBS1567			ARC182			MDL
	Found in solid	CRM	Recovery (%)	Found in solid	CRM	Recovery (%)	Found in solid	CRM	Recovery (%)	in solid
Ca	133.6	120	111.33	195	190	102.63	2670	2860	93.36	0.99
Cu	153.3	158	97.03	2.105	2	105.25	N/A	N/A	N/A	0.16
Fe	192.9	194	99.43	18.86	18.3	103.06	N/A	N/A	N/A	4.3
Mg	576.5	600	96.08	N/A	N/A	N/A	719.2	785	91.62	0.46
Mn	10.14	9.9	102.42	8.634	8.5	101.58	12.98	12.9	100.62	0.01
Ni	N/A	N/A	N/A	0.1719	0.18	95.5	0.2863	0.271	105.65	0.03
P	11490	11100	103.51	N/A	N/A	N/A	N/A	N/A	N/A	0.24
Zn	122.2	123	99.35	10.96	10.6	103.4	29.16	28.9	100.9	0.03

Conclusion

The results table show that major and trace elements were measured with equal success. Precise, accurate results for digested foodstuffs samples are easily attained on the Thermo Scientific iCAP 7400 ICP-OES Duo. The full wavelength coverage of the unique CID detector allowed the optimum wavelengths to be selected while the sensitivity of the Duo torch provided the lowest possible detection limits for this application.

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