Application Note: 40755

Food Series - Robust single method determination of major and trace elements in foodstuffs using the Thermo Scientific iCAP 6000 Series ICP

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Key Words

- ICP
- iCAP 6000 Duo
- Foodstuffs
- Micronutrients
- Microwave digestion
- Toxic elements

Benefits in brief

- Regulatory compliance assisted with online QC checking
- Single method for multiple matrices
- Wide dynamic range - ppb to % level in one method
- Sub ppb level detection limits achieved

Introduction

During the last decade, 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.

Instrumentation

The iCAP 6000 Series employs a high-resolution Echelle spectrometer with a much improved charge injection device (CID) detector. Advancements in CID technology allow this detector to feature higher sensitivity and lower noise than any of its predecessors. A Duo model 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 listed in Table 1 can be applied to an iCAP 6300 or 6500 as both are equally suited to this analysis.

PARAMETER	SETTING
Pump tubing	Orange/white tygon sample White/white tygon drain
Pump rate	50 rpm or 0.22 MPa
Nebulizer	Standard concentric
Nebulizer argon flow rate	0.6 L/min
Spray chamber	Standard cyclonic
Centre tube	2.0 mm
Torch orientation	Duo
RF forward power	1150 W
Auxiliary flow	0.5 L/min
Integration times High wavelengths Low wavelengths	 5 seconds 15 seconds

Table 1: Instrument Parameters.

Method

Reagents

- Nitric Acid, 69 %, AnalaR grade.
- Hydrochloric Acid, 37 % A.R. grade.
- 1000 ppm single element standards for As, Ca, Cu, Fe, Mg, Mn, Ni, P and Zn.

Certified Reference Materials

- Total Diet (ARC182)
- Bovine Liver (NBS1577a)
- Wheat Flour (NBS1567)

Sample Preparation

A 0.5 g aliquot of the Total Diet was digested in 5 ml HNO_3 and 1 ml HCl 0.5 g aliquots of the Wheat Flour and Bovine Liver were digested in 9 ml HNO_3 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 were used to prepare the calibration standards for this method. They were then acid matched to the samples. Table 2 indicates the concentration of each of the standards, selected to cover the linear range of the samples.

ELEMENT	CONCENTRATION IN PPM
As	0.01, 0.05
Cu, Mn	0.1
As, Ni	0.5
As, Cu, Fe, Mn, Ni, P, Zn, Ca, Mg	1
Zn	5
Cu, Fe, Ca, Mg, P	10
Ca, P	50

Table 2: Standard Concentrations.

ELEMENT VIEWING	
As,Cd, Cu, Mn, Ni, Pb, Zn	Axial
Ca, Fe, Mg	Radial

Table 3: Element Orientation.

Method development

Initially, more than one wavelength was selected for each element (using multiple views axial/radial). The subarrays for each wavelength were then examined (see example Figure 1) 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. 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. iTEVA's integrated Check Table functions allowed the introduction of QC standards and Limit Checks, which then automatically control the specifications of the method and flag samples for dilution.

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. (See Figure 2 for an example calibration) 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 below. 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.

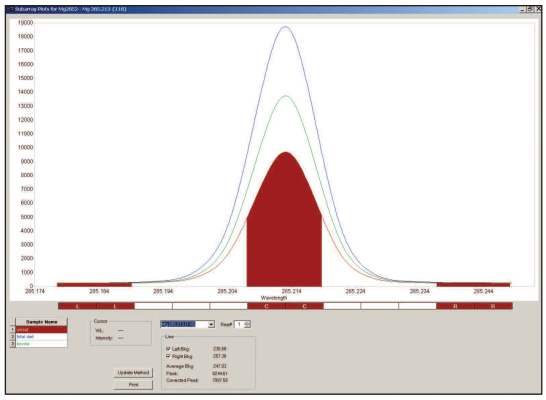

Figure 1: Subarray plot showing integration and background correction areas.

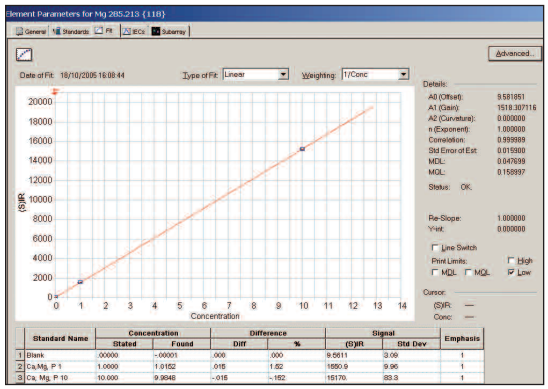

Figure 2: Calibration curve with readback of standards.

Conclusions

It is clear from the results table that the major and trace elements were measured with equal success. Precise, accurate results for digested foodstuffs samples are easily attained on the iCAP 6000 Series Duo ICP. The full wavelength coverage of the unique CID detector allowed the optimum wavelength to be selected while the sensitivity of the Duo torch provided the lowest possible detection limits for this application.

Element & Wavelength Units	NBS1577a Found ppm	NBS1577a Cert. mg/kg	NBS1577a % recovery	NBS1567 Found ppm	NBS1567 Cert. mg/kg	NBS1567 % recovery	ARC182 Found ppm	ARC182 Cert. mg/kg	ARC182 % recovery	MDL ppm
As 189.042 nm	0.0503	0.047	107.02	---	---	---	---	---	---	0.004
Ca 317.933 nm	133.6	120	111.33	195	190	102.63	2670	2860	93.36	0.0099
Cu 327.396 nm	153.3	158	97.03	2.105	2	105.25	---	---	---	0.0016
Fe 274.932 nm	192.9	194	99.43	18.86	18.3	103.06	---	---	---	0.0433
Mg 285.213 nm	576.5	600	96.08	---	---	---	719.2	785	91.62	0.0046
Mn 257.610 nm	10.14	9.9	102.42	8.634	8.5	101.58	12.98	12.9	100.62	0.0001
Ni 231.604 nm	---	---	---	0.1719	0.18	95.50	0.2863	0.271	105.65	0.0003
P 178.284 nm	11490	11100	103.51	---	---	---	---	---	---	0.0024
Zn 206.200 nm	122.2	123	99.35	10.96	10.6	103.40	29.16	28.9	100.90	0.0003

Table 4: Results and recoveries of Certified Reference Materials. Method Detection Limits (MDLs) are derived from liquid acid matched blank. % recovery is calculated on the stated value and does not make correction for statistical bias allowed.

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