Cost-effective analysis of major, minor and trace elements in foodstuffs using the 4100 MP-AES

Tran Nham and Craig Taylor, Agilent Technologies, Melbourne, Australia

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Introduction

Whether the goal is food safety, ensuring quality or establishing provenance, accurately measuring the trace element content of foods and beverages that we all consume is critical. While some elements are essential for our wellbeing at low concentrations, others like lead and chromium are highly toxic and more still are being linked to viral, neurological and other diseases. Food scares related to contamination or poor quality, not only constitute a health risk, they also undermine consumer confidence. This can lead to lost earnings through reduced sales and loss of credibility through adverse publicity.
Atomic spectroscopy is well established for the analysis of metals in foods and the technique employed often depends on the requirements of the application in terms of elements of interest, expected concentrations, and number and type of samples. Other important procurement factors that influence instrument selection include purchase and operational budget for consumables, gases, power and labour, as well as service and maintenance costs. Microwave Plasma-Atomic Emission Spectrometry (MP-AES) offers many advantages for food-testing facilities, including enhanced sensitivity, fast multi-element analysis capability, reduced running costs, and improved laboratory safety through the use of non-flammable nitrogen. This paper describes the analysis of a range of food and beverage samples using the Agilent 4100 Microwave Plasma Atomic Emission Spectrometer (MP-AES). This study shows that following a quick and simple acid digestion sample preparation procedure, a range of food samples including certified and standard reference materials, can be analyzed for trace and major element concentrations with good accuracy using MP-AES.

The Agilent 4100 MP-AES is a fast sequential atomic emission spectrometer that uses magnetically-coupled microwave energy to generate a robust and stable plasma using nitrogen gas. This stable plasma is capable of analyzing both aqueous and challenging organic matrices. This technique produces linear dynamic range, detection limits and analysis speed superior to conventional flame AAS. Based on an atomic emission technique, this elemental analysis technique also produces greater sensitivity than flame AAS. In addition, it eliminates the need to use hollow cathode lamps. This innovative elemental analysis technique also eliminates the need for argon or indeed, any bottled gas. It operates from a compressed air supply, using the Agilent 4107 Nitrogen Generator, producing a significant reduction in operating costs and reduced infrastructure costs. By using a nitrogen plasma, the 4100 MP-AES eliminates the need for expensive and dangerous gases, such as acetylene, resulting in lower running costs, unattended operation, and removing any safety concerns associated with use of acetylene and nitrous oxide when compared with traditional elemental analysis techniques like flame atomic absorption spectrometry.

Experimental

This work describes the analysis of various certified and standard reference materials per the sample descriptions below:

- NIES CRM No.7 Tea Leaves: from National Institute of Environmental Studies (NIES), Japan.
- NIES CRM No.10c Rice Flour: from National Institute of Environmental Studies (NIES), Japan.
- NIST SRM 1577 Bovine Liver: from National Institute of Standards and Testing, USA.
- CRM-Wheat Flour: from High Purity Standards, USA
- CRM-Milk Powder: from High Purity Standards, USA
- CRM-Oyster Tissue: from High Purity Standards, USA

Sample preparation

A simple acid digestion method was used to prepare three of the samples. Initially, 0.25 g of the tea leaves CRM, 0.5 g of bovine liver SRM and 1 g of rice flour CRM were weighed into separate 250 mL beakers. This was then followed by the addition of 10 mL of HNO3 and each beaker was covered with a watch glass. The samples were heated on a hot plate until completely dissolved. After cooling to room temperature, each digest was transferred to a 100 mL volumetric flask and made up to the required volume by adding Milli-Q water.
Pre-prepared sample solutions of CRM-Wheat Flour, CRM-Milk Powder and CRM-Oyster Tissue in 4% HNO3 were purchased from High Purity Standards, USA.
Working standards and a blank were matrix-matched with the samples.

Experimental

Instrumentation

The innovative 4100 MP-AES with its proprietary Microwave Excitation Assembly is a sequential atomic emission spectroscopic technique capable of fast, unattended multi-element analysis at varying concentration levels using a nitrogen plasma. The unique Microwave Excitation Assembly focuses and contains the microwave energy that is created via a concentrated axial magnetic field around the torch. This creates a robust toroidal plasma that allows the stable introduction of liquid samples.
With a central channel temperature of ~5,000 K, MP-AES is highly suited to spectroscopic analysis, as it creates high intensity atomization emission lines. In addition to simplified spectra, nitrogen-MP-AES offers reduced operating costs and increased lab safety compared to flame AA, through the avoidance of costly and highly flammable gases such as acetylene.
The analysis was carried out using an Agilent 4100 MP-AES equipped with a standard MP-AES torch, concentric nebulizer, and glass cyclonic spray chamber.
Operating parameters are shown in Table 1.

Table 1. Agilent 4100 MP-AES Operating Parameters

Instrument Parameter	Setting
Nebulizer pressure	160-180 kPa
Read time	3 s (10 s for MDL)
No. of replicates	3, (10 for MDL)
Stabilization time	15 s
Background correction	Auto

Results and Discussion

Method detection limits

The Method Detection Limits were determined from the analysis of digested blank samples. The selected analytical wavelengths and method detection limits (3σ) are listed in Table 2.

Table 2. Agilent 4100 MP-AES element wavelength and method detection limits (ppb)

Element	Wavelength (nm)	MDL (µg/kg)
Al	396.152	0.5
Ba	455.403	0.02
Ca	445.478	14
Cd	228.802	1.2
Co	340.511	4
Cr	425.433	0.5
Cu	327.396	0.4
Fe	371.993	3
K	769.897	3
P	213.618	100
Pb	405.781	5
Mg	518.361	4
Mn	403.076	0.5
Mo	379.825	1.5
Na	589.592	3
Ni	341.476	2
Ni	352.453	2
Sr	407.771	0.01
Zn	213.857	4

Analysis of foodstuffs

Results of the analysis of major, minor and trace extractable elements in six different foodstuffs are listed in Tables 3 to 8. The measured values (carried out in triplicate) are in good agreement with the certified values for all CRM and SRM samples.

Results and Discussion

Table 3. Results of NIES No.7 Tea Leaves

Element	Measured values	Certified values
	wt%	wt%
Ca	0.314 ± 0.013	0.320 ± 0.012
Mg	0.150 ± 0.004	0.153 ± 0.006
K	1.861 ± 0.074	1.86 ± 0.07
	mg/kg	mg/kg
Al	686 ± 15	775 ± 20
Ba	5.76 ± 0.57	5.7
Cd	nd	0.03 ± 0.03
Co	nd	0.12
Cr	nd	0.15
Cu	7.13 ± 0.81	7 ± 0.3
Pb	nd	0.8 ± 0.03
Mn	631.4 ± 4.9	700 ± 25
Ni	6.03 ± 0.63	6.5 ± 0.3
Sr	3.63 ± 0.43	3.7
Zn	34 ± 3	33 ± 3

Table 4. Results of NIES No.10c Rice Flour

Element	Measured values	Certified values
	wt%	wt%
Mg	0.127 ± 0.006	0.125 ± 0.008
K	0.279 ± 0.012	0.275 ± 0.010
P	0.300 ± 0.010	0.335 ± 0.008
	mg/kg	mg/kg
Al	1.49 ± 0.13	1.5
Ca	95.4 ± 7.0	95 ± 2
Cd	1.83 ± 0.14	1.82 ± 0.06
Co	nd	0.007
Cr	nd	0.08
Cu	4.03 ± 0.32	4.1 ± 0.3
Fe	10.55 ± 0.15	11.4 ± 0.8
Mn	34.1 ± 0.8	40.1 ± 2.0
Mo	nd	1.6 ± 0.1
Ni	nd	0.30 ± 0.03
Sr	0.2	0.2
Zn	21.8 ± 1.0	23.1 ± 0.8

Table 5. Results of NIST 1577 Bovine Liver

Element	Measured values	Certified values
	wt%	wt%
Na	0.247 ± 0.006	0.243 ± 0.013
K	1.00 ± 0.08	0.97 ± 0.06
	mg/kg	mg/kg
Ca	130.7 ± 5	123
Cd	nd	0.27 ± 0.04
Co	nd	0.18
Cu	184.7 ± 6.1	193 ± 10
Fe	266 ± 5	270 ± 20
Pb	nd	0.34 ± 0.08
Mg	625 ± 45	605
Mn	10.35 ± 1.41	10.3 ± 1
Mo	nd	3.2
Sr	0.15 ± 0.07	0.14
Zn	125.1 ± 4.3	130 ± 10

Table 6. Results of CRM-Wheat Flour.

Element	Measured values mg/kg	Certified values mg/kg
Al	0.83 ± 0.02	0.85 ± 0.01
Ca	9.64 ± 0.97	9.5 ± 0.1
Cd	nd	0.0015
Co	nd	0.001
Cr	0.013 ± 0.001	0.014
Cu	0.09 ± 0.008	0.1 ± 0.002
Fe	0.81 ± 0.04	0.90 ± 0.01
K	62.5 ± 0.5	65 ± 0.7
P	61.1 ± 1.7	65 ± 0.7
Pb	0.05 ± 0.001	0.050 ± 0.003
Mg	20.8 ± 0.1	20.0 ± 0.2
Mn	0.36 ± 0.02	0.4 ± 0.008
Ni	nd	0.009 ± 0.001
Zn	0.47 ± 0.05	0.50 ± 0.01

Table 7. Results of CRM-Milk Powder

Element	Measured values mg/kg	Certified values mg/kg
Al	nd	0.020 ± 0.002
Ca	130.9 ± 9	130 ± 1
Co	nd	0.0004
Cr	nd	0.0003
Cu	0.006 ± 0.001	0.007 ± 0.001
Fe	0.018 ± 0.002	0.020 ± 0.001
K	178 ± 6	170 ± 2
P	98.7 ± 1.3	100 ± 1
Pb	nd	0.002
Mg	11.9 ± 0.2	12 ± 0.1
Mn	0.003 ± 0.002	0.003
Na	48.7 ± 2.6	50 ± 1
Zn	0.48 ± 0.05	0.50 ± 0.01

Table 8. Results of CRM-Oyster Tissue

Element	Measured values mg/kg	Certified values mg/kg
Al	2.92 ± 0.07	3
Ca	15.03 ± 0.49	15
Cd	nd	0.03
Co	nd	0.004
Cr	nd	0.007
Cu	0.56 ± 0.05	0.6
Fe	1.73 ± 0.07	2
K	100.4 ± 0.96	100
P	79.1 ± 0.9	80
Pb	nd	0.005
Mg	12.05 ± 0.2	12
Mn	0.18 ± 0.01	0.2
Na	48.9 ± 0.8	50
Ni	nd	0.01
Zn	8.3 ± 0.4	9

Conclusions

The Agilent 4100 MP-AES offers any food testing facilities dependent on acetylene-based instrumentation a real alternative in terms of sensitivity, multi-element capability and speed of analysis, while cutting operating costs and improving the safety of the lab environment through the use of non-flammable nitrogen.
This study shows that following a quick and simple acid digestion sample preparation procedure (required for three of the six diverse food samples), all six certified and standard reference materials can be analyzed for trace and major element concentrations with good accuracy by MP-AES.
The addition of the Agilent 4107 Nitrogen Generator is also possible in order to perform this analysis with significantly lower gas costs or for analysis in remote locations where sourcing of gases is costly or difficult.