Analysis of Chinese herbal medicines by microwave plasma-atomic emission spectrometry (MP-AES)

Application note

Food Testing

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Introduction

Traditional Chinese herbal medicines (TCMs) and their related products have been widely used in China for centuries and the Chinese government, as well as academic research scientists, are paying greater attention to the safety issues of TCMs in clinical use.

With recent developments in science and technology, there is a greater awareness of heavy metals such as Pb, Cr, Cu, Ni, As, K, Na and Ca, which are present in some TCMs. Therefore the analysis of both toxic and beneficial heavy metals is crucial in quality control of TCMs, and regulations have been implemented to restrict their levels. In order to enhance TCM safety and management, the Chinese Pharmacopoeia (2010 edition) was created in July 2010 and this is now the standard for Chinese medicinal crops, Chinese herbal pieces, Chinese patent drugs, and herbal extracts.

The aim is to control the content of heavy metals and toxic elements in traditional Chinese medicine, and limits were created for various products such as medical injections, the fruit of Chinese wolfberry, ginseng, dangshen, hawthorn, and so forth, as well as those suitable for children and medicines used for prolonged periods. The export of TCMs is under intense scrutiny due to strict regulations, and the high content of heavy metals in traditional Chinese medicine has resulted in an international response. By nature TCMs are very complex and contain many elements at different concentration levels — both high and low.

A rapid, simple and accurate method has been developed for the determination of key elements in Chinese herbal medicine samples using a new elemental analysis technique — microwave plasma-atomic emission spectrometry (MP-AES). MP-AES provides an ideal, low-cost solution for multi-element analysis of TCMs, with excellent long-term stability, reduced running costs, and improved lab safety.

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 innovative elemental technique produces linear dynamic range, detection limits and analysis speeds superior to conventional flame AAS. 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 the use of acetylene and nitrous oxide. In addition the 4100 MP-AES eliminates the need to use hollow cathode lamps and when used in conjunction with the Agilent 4107 Nitrogen Generator, operating costs are significantly reduced.

The results were in good agreement with traditional techniques. The recoveries of the method were between 90 and 110% and the method detection limits for most elements were less than 3 µg/mL in the solid sample. The recovery and precision were also excellent, demonstrating the ability of the 4100 MP-AES to meet the needs of a very demanding industry.

Experimental

Instrument

An Agilent 4100 MP-AES was used for the total metal determination of a range of analytes in herbal medicines. The viewing position and nebulizer pressures were optimized automatically using the Agilent MP Expert software. Table 1 lists the instrument conditions.

Table 1. Instrument conditions

Instrument parameter	Setting
Nebulizer	Glass concentric
Spray chamber	Single-pass glass cyclonic
Sample tubing	Orange/green
Waste tubing	Blue/blue
Read time	3 s
Nebulizer pressure	160–220 kPa
Number of replicates	3
Stabilization time	15 s
Background correction	Auto

Sample preparation

The samples were prepared by microwave digestion. Approximately 0.40 g samples of West Gan leaf, Endogenous galanin leaf and Ginseng were weighed and placed into a PTFE vessel. Samples were prepared in duplicate using 5 mL HNO_3, 0.5 mL HCL and 1 mL H_2O_2. After the digestion, the vessel was cooled to room temperature before dilution to a final volume of 30 mL.

Results and discussion

Calibration curves

The calibration was prepared using a multi-element standard solution in a matrix of 5% HNO_3. Linear correlation coefficients for all analytes were better than 0.999. Figure 1 shows some typical elements calibration curves.

Sample results

Three typical herbal medicines were analyzed using the method described and the results compared with traditional analysis techniques. The results obtained for the MP-AES are in strong agreement with the traditional analysis techniques, as shown in Table 2.

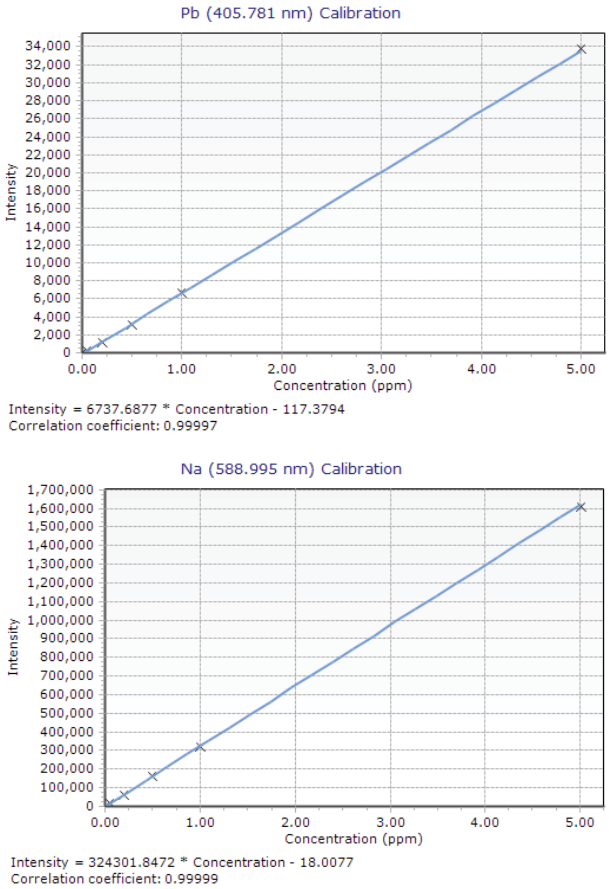

Figure 1. Typical element calibration curves

Method detection limit

The blank sample was analyzed 11 times under optimized conditions. The method detection limits (MDLs) for each element were calculated and can be seen in Table 3.

Table 3. The method detection limits as ppb in sample

Element	MDL (ppb)
Al	0.2
Ba	0.05
Ca	0.02
Cr	0.1
Cu	0.07
Fe	0.35
K	0.05
Mg	0.03
Mn	0.06
Na	0.03
Ni	0.5
P	2.9
Pb	0.6
Zn	0.4

Table 2. Sample results in µg/mL

Sample element	West Gan leaf		Endogenous galanin leaf		Ginseng	
	MP-AES	Traditional techniques	MP-AES	Traditional techniques	MP-AES	Traditional techniques
Al	420	425	411	418	294	289
Ba	47.56	47.52	17.75	17.72	42.12	41.61
Ca	18622	18659	13247	13305	2168	2212
Cr	22.62	22.95	5.88	5.96	1.16	1.06
Cu	9.07	9.04	6.75	6.80	6.17	6.16
Fe	820	821	457	458	193	199
K	18510	18651	17797	17829	11374	11220
Mg	5065	5018	4973	4951	1296	1337
Mn	297	299	372	371	33.47	33.76
Na	45.75	45.90	57.75	56.71	252	243
Ni	1.40	1.58	1.61	1.63	1.68	1.65
P	3579	3588	2030	2030	2296	2245
Pb	-	-	1.25	1.21	-	-
Zn	29.85	29.75	40.08	39.46	19.43	20.07

In order to confirm the method, the National Standard Substances (leaves of aspen) GBW07604 was prepared and analyzed at the same time. As this sample is also a type of leaf, it has similar characteristics to most types of Chinese herbal medicines and is often used for quality control of TCM analysis. As can be seen in Table 4, the measured values are in good agreement with the certified values.

Table 4. Results of GBW07604

Element	Unit	Certifed	MP-AES
Al	%	0.104 ± 0.06	0.06
Ba	µg/g	26 ± 4	25
Ca	%	1.81 ± 0.13	1.68
Cr	µg/g	0.55 ± 0.07	0.55
Cu	µg/g	9.3 ± 1	9.0
Fe	µg/g	274 ± 17	269
K	%	1.38 ± 0.07	1.35
Mg	%	0.65 ± 0.05	0.66
Mn	µg/g	45 ± 4	49
Na	µg/g	200 ± 13	188
Ni	µg/g	1.9 ± 0.3	1.7
P	µg/g	1680 ± 60	1720
Zn	µg/g	37 ± 3	39

Conclusions

The Agilent 4100-MP-AES has been shown to be an ideal solution for the analysis of heavy metals in traditional Chinese herbal medicines (TCMs). The study shows that preparing samples by microwave digestion and subsequent analysis by 4100 MP-AES, three typical TCMs can be analyzed for trace and major concentration elements with good accuracy. Furthermore, the Agilent 4100 MP-AES has the lowest operating costs of comparable techniques such as flame AA, and by using non-flammable gases, removes safety concerns associated with acetylene and nitrous oxide.

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 or where sourcing of gases is costly or difficult.

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Published March 23, 2012
Publication number: 5990-9791EN

Agilent Technologies