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Application News

No.J97

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Herbal Medicines by ICPE-9000

Introduction

The use of herbal medicines as an alternative to traditional western medicine has been increasing, and consequently this trend has been accompanied by heightened interest and concern regarding the quality and safety of herbal medicines. Herbal medicines—medicinal products that naturally contain a variety of flora and fauna, bacteria, minerals, etc—are currently utilized as medications (over-the-counter drugs), foods, functional foods, and dietary supplements without purification of their active ingredients. Herbal medicines that are used as pharmaceuticals are subject to standards associated with their behavior, chemical properties, and safety as specified in official regulations, including the Japanese Pharmacopoeia. Safety is assessed using limit testing, including heavy metals testing and arsenic limit testing. However, since the biological effects of heavy metals vary depending on the metal, testing of metal content is required for each metal. Here, we present the results of our analysis of herbal medicines using the Shimadzu ICPE-9000 multi-type ICP emission spectrometer.

Samples

The samples consisted of herbal medicines that are readily available in Japan.

Sample Preparation

As acidification and heating will cause volatilization of low-boiling-point elements such as arsenic (As) and mercury (Hg), a pretreatment method that permits efficient dissolution of the sample with minimal loss of these elements is required. Here, we employed microwave assisted acid Closed Vessel digestion method for pretreatment.
For the digestion, 7.5 mL of concentrated nitric acid and 0.5 mL of concentrated hydrochloric acid were added to 0.5 g of dried sample, which was then transferred to a microwave digestion system (ETHOS One microwave sample preparation system, from Milestone General K.K.).
Following the digestion, ultrapure water was added to the processed solution to bring the volume to 25 mL, and this was used as the analytical sample. Separately, a sample was prepared consisting of standard solution spiked with the analyte elements for use in spike and recovery testing.

Analytical Instrument

Herbal medicines are said to number in the hundreds, and elemental analysis of these medicines must be conducted accurately and efficiently. The ICPE-9000 is a high-throughput, high-performance instrument that features ease of use, low running cost, high sensitivity, and low contamination.

Table 1 Analytical Conditions

Instrument	: ICPE-9000
Radio frequency power	: 1.2 kw
Plasma gas	: 10 L/min
Auxiliary gas	: 0.6 L/min
Carrier gas	: 0.7 L/min
Sample introduction	: Coaxial nebulizer
Misting chamber	: Cyclone chamber
Plasma torch	: Mini torch
View direction	: Axial

Analysis

Quantitation was conducted by the calibration curve method using the ICPE-9000. Elemental analysis was conducted for arsenic, cadmium, chromium, copper, mercury, lead, and tin; all elements that are considered to have relatively high oral toxicity.

Results

Table 2 shows the semi-quantitation results (wt-%) for the principal components determined in qualitative analysis. The ICPE-9000 acquires and saves the qualitative data for all elements at the time of quantitative analysis. The semi-quantitative concentrations are calculated automatically from the values stored in the software-integrated database.
Herbal medicines often contain large amounts of calcium, potassium, and magnesium as coexisting substances. It is not uncommon for errors to occur in analytical values due to ionization interference and other factors when samples contain large amounts of coexisting components. In this regard, and compared to the typical torch, the high-temperature plasma achieved with the mini-torch adopted in the ICPE-9000 permits high sensitivity while suppressing the adverse effects of ionization interference.
Table 3 shows the quantitation results, recovery rates, and detection limits. Excellent recovery results were achieved for all elements, and measurement was conducted without any interference from the principal components. Moreover, measurement was clearly conducted with sufficient sensitivity, and all the detection limits were below the specified Japanese Pharmacopoeia limit values, as well as the Chinese import/export standard values.
The measurement cycle is very fast, taking only about two and a half minutes per sample, including the time required for sample introduction and rinse. Furthermore, use of the autosampler permits automated measurement of multiple samples, permitting even greater efficiency.

Table 2 Semi-Quantitative Results for Herbal Medicines by Qualitative Analysis

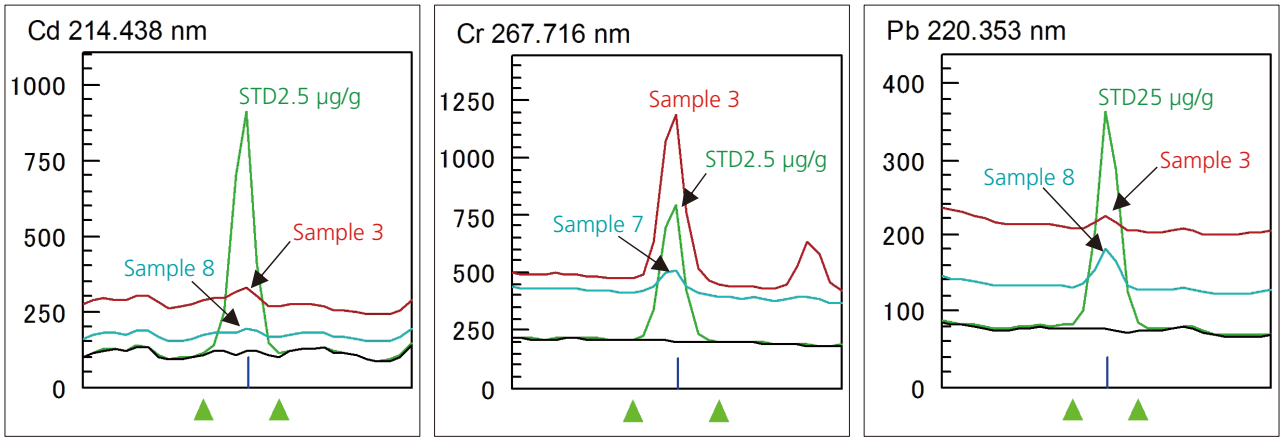
	Ca	K	Mg	S	Al	P	Si	Fe	Mn	Ba	Sr	Na
Horny goat weed	2.6	1.0	0.4	0.3	0.2	0.2	0.14	0.13	0.05	0.02	0.01	0.01
Fang feng (Saposhnikoviae Radix)	1.2	0.5	0.4	0.2	0.2	0.2	0.08	0.10	0.004	0.01	0.02	0.08

Table 3 Quantitative Results for Herbal Medicines (µg/g)

	Element Name	As	Cd	Cr	Cu	Hg	Pb	Sn
	*Detection Limit	0.2	0.005	0.02	0.04	0.1	0.1	0.05
	Sample Name							
1.	Cardamom	<	0.07	<	5.5	<	0.2	<
2.	Cinnamon	0.3	<	0.5	7.0	<	0.5	<
3.	Horny goat weed	0.5	0.14	3.0	4.7	<	1.4	0.1
	Recovery Rate in Spike/Recovery Test (%)	101.1	98.5	98.8	95.2	97.9	95.2	100.3
4.	Carrot	<	0.03	0.04	5.2	<	<	<
5.	Rehmanniae radix	<	<	0.3	3.9	<	0.2	<
6.	Paeoniae radix	<	<	0.2	4.3	<	<	0.1
7.	Fang feng (Saposhnikoviae Radix)	<	<	0.4	7.0	<	0.2	<
	Recovery Rate in Spike/Recovery Test (%)	100.9	101.7	98.7	100.1	97.4	97.6	99.6
8.	Turmeric (Curcumae Radix)	<	0.05	0.1	2.2	<	4.5	<
Japanese Pharmacopoeia Limit Value (varies with sample)		2 – 5	10 – 15 (heavy metal test: lead conversion)					
WHO Recommended Level			0.3				10	
People's Republic of China Herb Import/Export Standard Values		2	0.3		20	0.2	5	

* Detection limit : Detection limit (3σ) determined from standard deviation using N = 5 repeat measurements of calibration curve blank

* < : Below the limit of detection



* The concentrations in the figures refer to the concentrations in the samples (solid).

Fig. 1 Spectral Profiles of Cd, Cr, and Pb in Herbal Substances

[References]
1) Sixteenth Edition of Japanese Pharmacopoeia (edited by Society of Japanese Pharmacopoeia)
2) WHO Guidelines for Assessing Quality of Herbal Medicines with Reference to Contaminants and Residues (issued March 2009 by Japan Self-Medication Industry)
3) Green Trade Standards of Importing & Exporting Medicinal Plants & Preparations (issued April 2001 and enacted July 1, 2001 by Ministry of Foreign Trade and Economic Cooperation, the People's Republic of China)

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