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Application News

No.J101A

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Harmful Elements in Herbal Medicines by ICPE-9800 Series

Introduction

Herbal medicines, products consisting of animals and plants, fungi, and minerals with naturally occurring efficacy, are used without purification as drugs (over-the-counter drugs), foods, functional foods, and dietary supplements. Safety standards for harmful elements used in pharmaceuticals are provided for in each country. Table 1 shows examples of regulations regarding the permissible levels of harmful elements in herbal medicines, including the levels suggested by WHO[1] and the import/export reference values of China[2].

Here, using the Shimadzu ICPE-9800 series multi-type ICP atomic emission spectrometer, we conducted an analysis of an herbal medicine. The ICPE-9800 series, with its mini-torch plasma and spectrometer which permits all element/all wavelength simultaneous analysis, provides high sensitivity, high precision, and high throughput at low cost.

Table 1 Harmful Element Regulation of Herbal Medicines (mg/kg)

Element	As	Cd	Cu	Hg	Pb
WHO recomended value		0.3			10
China Import/Export Herbal Medicine Reference Value	2	0.3	20	0.2	5

Sample

Herbal medicines distributed in Japan.

Sample Preparation

Low boiling point-elements such as arsenic (As), mercury (Hg), etc. are subject to loss due to volatilization during operations such as heating and acid addition. Therefore, a pretreatment process is required that will keep element loss to a minimum, while at the same time be efficient. Here, we conducted sample digestion using a microwave sample preparation system.

To 0.5 g of dry sample, 7.5 mL of concentrated nitric acid and 0.5 mL of concentrated hydrochloric acid were added, and digestion was conducted using a microwave sample preparation system. After digestion, distilled water was added to the process liquid to bring the total volume to 25 mL, and this was used as the analytical sample. Separately, after preparing another sample in the same manner, a standard solution containing the target elements was added, and this served as the spike-and-recovery test solution.

Instrument and Analytical Conditions

For measurement, the Shimadzu ICPE-9800 series multi-type ICP emission spectrometer was used. The analytical conditions used are shown in Table 2. The adoption of an echelle spectrometer and a CCD detector permits simultaneous analysis of all the elements at all the wavelengths, in addition to high-throughput measurement even with many target elements and samples. Also, the mini-torch plasma, Eco mode, and vacuum-housed spectrometer serve to greatly reduce running costs due to gas consumption.

Herbal medicines often contain large amounts of coexisting elements, including calcium (Ca), potassium (K), and magnesium (Mg). Typically, when a sample contains many coexisting elements, some level of error may affect the analysis value due to ionization interference. However, the ICPE-9800 series mini-torch produces a high-temperature plasma which in addition to providing high sensitivity, also suppresses the effect of ionization interference.

Table 2 Analytical Conditions

Instrument	: ICPE-9800 series
Radio Frequency Power	: 1.2 kW
Plasma Gas Flowrate	: 10 L/min
Auxiliary Gas Flowrate	: 0.6 L/min
Carrier Gas Flowrate	: 0.7 L/min
Sample Introduction	: Nebulizer 10
Misting Chamber	: Cyclone chamber
Plasma Torch	: Mini Torch
Observation	: Axial (AX)
Measurement Time	: 2.5 min/sample (Including rinse time)

Analysis

Quantitative analysis of As, Cd, Cr, Cu, Hg, Pb, and Sn was conducted using the calibration curve method.

[References]

1) WHO Guidelines for Assessing Quality of Herbal Medicines with Reference to Contaminants and Residues (Japan Self-Medication Industry, published March 2009)
2) Green Trade Standards of Importing & Exporting Medicinal Plants & Preparations (Ministry of Foreign Trade and Economic Cooperation, People's Republic of China, effective July 1, 2001)

Analytical Results

Table 3 shows the semi-quantitative results for the principal components using qualitative analysis. Semi-quantitative results are calculated automatically by the database built into the software. With the ICPE-9800 series, qualitative data for all elements are acquired and saved at the same time that quantitative analysis is conducted. This feature, even after quantitative analysis, makes it possible to identify the principal elements and their concentrations, making it possible to consider the impact on the analyte elements. Table 4 shows the analytical results, and Table 5 shows the results of spike-and-recovery test. Fig. 1 shows the spectral profiles. The detection limit is below the WHO recommended value as well as the China import and export reference values, indicating sufficient sensitivity. Further, as for the spike-and-recovery rate, good results were shown for all elements, and it is clear that accurate quantitation is possible without adverse influence from coexisting elements such as Ca and K present at high concentrations.

Conclusion

Use of the ICPE-9800 series offers high sensitivity, as well as accurate and low-cost measurement of harmful elements in herbal medicines.

Table 3 Semi-Quantitative Results for Herbal Medicines by Qualitative Analysis (wt%)

	Ca	K	Mg	S	Al	P	Si	Fe	Mn	Ba	Sr	Na
Horny goat weed	3.7	1.1	0.35	0.45	0.18	0.31	0.16	0.12	0.060	0.021	0.021	0.008
Fang feng (Saposhnikoviae Radix)	1.6	0.65	0.38	0.35	0.25	0.25	0.11	0.09	0.006	0.013	0.025	0.070

Table 4 Analytical Results for Herbal Medicines (µg/g)

Element / Sample Name	As	Cd	Cr	Cu	Hg	Pb	Sn
1. Cardamom	<	0.06	<	5.4	<	0.3	<
2. Cinnamon	<	<	0.4	6.6	<	0.6	<
3. Horny goat weed	0.4	0.13	2.8	4.5	<	1.5	<
4. Carrot	<	0.03	0.04	5.0	<	<	<
5. Rehmanniae Radix	<	<	0.4	3.8	<	<	<
6. Paeoniae radix	<	<	0.3	4.2	<	<	<
7. Fang feng (Saposhnikoviae Radix)	<	<	0.5	6.6	<	<	<
8. Turmeric (Curcumae Radix)	<	0.04	0.1	2.1	<	4.3	<
Detection Limit	0.2	0.007	0.02	0.04	0.07	0.1	0.04
WHO Recommended Value		0.3				10	
China Import/Export Herbal Medicine Reference Value	2	0.3		20	0.2	5	

Detection limit: 3 times the concentration standard deviation obtained from 10 repeated measurements of the calibration curve blank × Dilution factor (50)

<: Below the limit of detection

Table 5 Recovery Rate in Spike/Recovery Test (%)

3. Horny goat weed	99	98	99	101	101	96	98
7. Fang feng (Saposhnikoviae Radix)	100	97	99	102	102	98	100

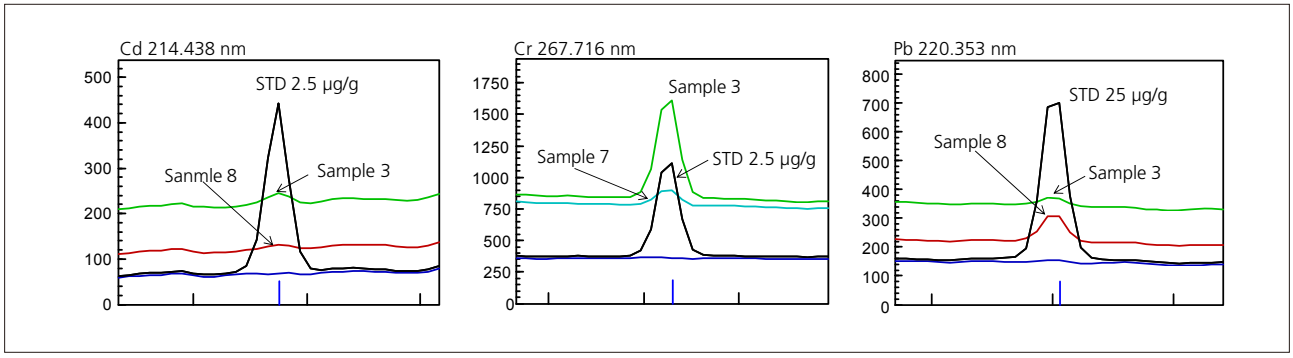

Fig. 1 Spectral Profiles of Cd, Cr, and Pb in Herbal Substances
*** The concentrations in the figures refer to the concentrations in the samples (solid)**

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