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Quantitative determination of heavy metals in ayurvedic tablets using ICP-MS

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Introduction

Ayurvedic medicine originated in India more than 2000 years ago and relies heavily on ayurvedic products. Approximately 80% of Indian population makes use of Ayurveda through more than one-half million Ayurvedic practitioners working in Ayurvedic hospitals and clinics. Heavy metals are considered as possible human carcinogen by the World Health Organization's International Agency if it exceeds the permissible limit. Some ayurvedic products do contain toxic materials such as heavy metals, pesticides, etc. Exposure to heavy metals such as Arsenic (As), Cadmium (Cd), Lead (Pb), and Mercury (Hg) can cause adverse health effects and toxicity.
The objective of this study is to develop a sensitive, selective, accurate and reliable method using Shimadzu ICPMS-2030 to determine the heavy metals from ayurvedic tablets.

Figure 1 Ayurvedic plant resources and tablets

Methods and Materials

Commercially available ayurvedic tablets (Figure 1) were used for the extraction study of heavy metals. The samples were digested using Anton Paar microwave digestion system. as per the programme Table 1. Pre-spiked recoveries were established by spiking samples with standard solution of heavy metals followed by subsequent analysis.

Microwave digestion:

Sample pretreatment procedure by microwave digestion

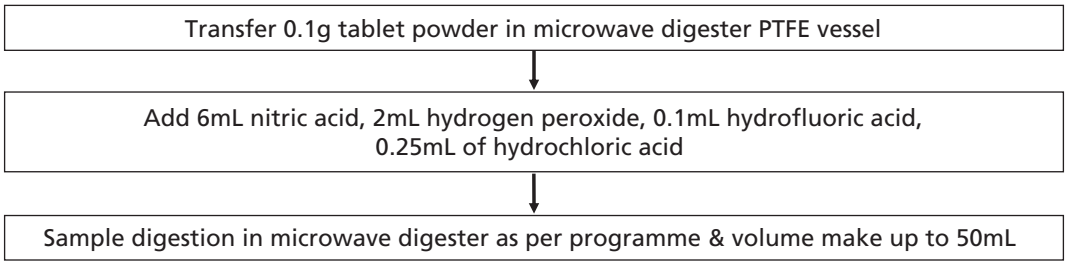

Table 1: Microwave digester programme

Steps	Ramp (min)	Temp (°C)	Hold time (min)
1	10	100	05
2	10	150	10
3	10	180	10
4	10	200	10

Figure 2 Shimadzu ICPMS-2030 Inductively coupled plasma mass spectrometer

Results

Method development

Sample preparation is very crucial and important step in elemental analysis. ayurvedic drugs contain both high amount of organic and inorganic components which are very difficult to break. A robust sample preparation method was developed by using combination of acids to digest the sample completely. Pre-spiked std. recovery study was performed to check the extraction method and matrix interference.

Quantification of heavy metals (As, Cd, Pb and Hg) was performed using internal standard method by plotting calibration curves for respective elements. Best elemental masses were selected for analysis based on their isotopic ratio, background equivalent concentration (BEC) values and isobaric interference. The internal standard was selected for elements according to the best possible match in terms of proximity with respect to mass to charge ratio and ionization behavior. It was added to standards and samples at a concentration of 1µg/L in final volume. ICPMS LabSolutions software special features like "qualitative analysis and development assistant" were used for auto method optimization. Also "quantitative analysis and diagnosis Assistant" were used for auto corrected report generation.

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Analytical Conditions (ICPMS-2030)	
Torch	: Mini torch
Radiofrequency	: 1.2 kW
Sampling depth	: 5 mm
Plasma gas (L/min)	: 8.0
Auxiliary gas (L/min)	: 1.1
Carrier gas (L/min)	: 0.7
Cell voltage	: -21 V
Cell gas (mL/min)	: 6.0
Energy Filter	: 7V
Chamber temp.	: 5 ˚C
Number of scans	: 10
Integration time	: 2sec
Peristaltic pump speed	: 60 r.p.m.- High 20 r.p.m.- Low
Isotopes monitored	= ^{75}As, ^{111}Cd, ^{208}Pb, ^{202}Hg
Internal standard	= ^{89}Y

Figure 3 shows standard calibration linearity curves in the concentration range of 0.5µg/L to 50µg/L. Results of regression analysis on calibration curves were shown in Table 2.

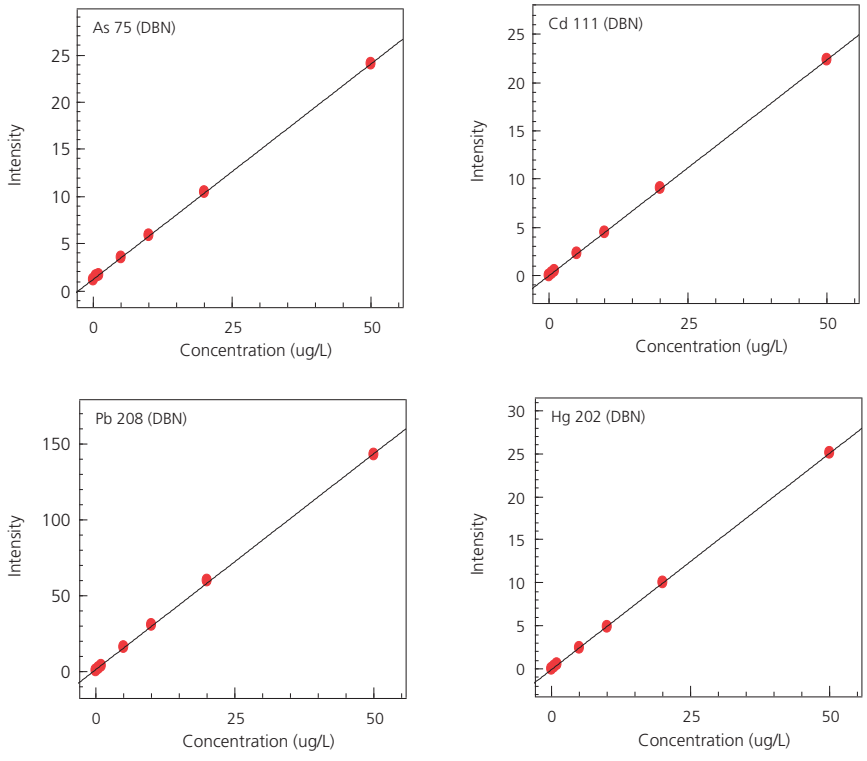

Figure 3 Standard calibration linearity curves 0.5µg/L to 50µg/L

Table 2 Results of regression analysis on calibration curves by ICPMS

Elements	Correlation coefficient (r)	LOD (3s) (µg/L)	LOQ (10s) (µg/L)
As	0.99999	0.2254	0.7515
Cd	0.99990	0.0009	0.0029
Pb	0.99983	0.0368	0.1227
Hg	0.99995	0.0099	0.0333

Quantitative Results

The quantitative analysis of As, Cd, Pb, Hg elements from ayurvedic tablets was performed by ICPMS-2030 and results obtained are shown in Table 3.
Recovery study was done by spiking respective sample with standard elements and analyzing the same under similar analytical conditions.
The results obtained were evaluated for statistical parameters like accuracy and linearity. Accuracy in terms of recovery was found to be between 80% to 120% for pre-spiked samples at level of 1µg/L & 5µg/L and %RSD obtained for all standards and samples were less than 6 as shown in Table 4. Linearity with co-relation coefficient greater than 0.999 was achieved.

Table 3 Average elemental results of ayurvedic tablets (n = 6 replicates)

Elements	Ayurvedic tablet (µg/L)	%RSD
As	89.06	4.89
Cd	5.00	3.76
Pb	115.75	4.84
Hg	6.20	2.34

Table 4 Average accuracy at 1.0µg/L & 5.0µg/L in ayurvedic tablets (n = 6 replicates)

Elements	1µg/L		5µg/L	
	%Recovery (Accuracy)	%RSD	%Recovery (Accuracy)	%RSD
As	92.67	3.02	98.63	5.07
Cd	91.37	2.79	87.00	2.38
Pb	-	-	83.00	3.73
Hg	92.87	3.68	85.97	2.93

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Conclusions

Heavy metals in ayurvedic tablets at ppb level were quantitatively estimated using ICPMS.
The proposed ICP-MS method has been proved to be a accurate and sensitive for detection of heavy metals in ayurvedic products.
A new “development assistant” function of LabSolutions software helped in setting analysis method (like linearity level, internal standard).

Disclaimer: Shimadzu ICPMS-2030 and application in this poster are intended for Research Use Only (RUO). Not for use in diagnostic procedures. Not available in the USA, Canada and China.

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