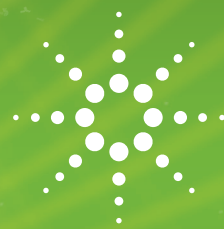


FOOD ANALYSIS

ANALYSIS OF TRACE LEVEL OF HEAVY METALS IN NOODLES AND FLAVOR SACHET USING THE AGILENT 7900 ICP-MS

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ABSTRACT

This solution note focuses on the analysis of toxic heavy metals in noodles and flavor sachets using the Agilent 7900 ICP-MS, in accordance with the regulatory requirements of the Food Safety Standards Authority of India (FSSAI).

Agilent's 7900 ICP-MS offers a complete solution, with excellent accuracy and precision of results. The food standard reference material, SRM 1568b, was analyzed for heavy metals and good recoveries were observed.

INTRODUCTION

With development and changing life styles, the fast food industry is growing at an exponential rate, leading to an increase in the consumption of ready-to-eat foods. The presence of heavy metals in these products has become a matter of growing concern, not only for the regulatory agencies but also for consumers. The Food Safety and Standards Authority of India (FSSAI) has been created for laying down science based food standards and to regulate their manufacture, storage, distribution, sale and import to ensure the availability of safe and wholesome food for human consumption. Four elements, arsenic, cadmium, mercury and lead, are strictly monitored and their maximum permissible limits are shown in Table 1.



Element	Limits, mg/kg
As	1.1
Cd	1.5
Hg	1.0
Pb	2.5

Table 1. Heavy Metal Limits as per FSSAI Regulations.



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ANALYTICAL TECHNIQUE

Instrumentation

An Agilent 7900 ICP-MS with 4th generation Collision/Reaction Cell (CRC), the Octopole Reaction System (ORS4) and standard sample introduction system (Nickel cones, glass concentric nebulizer, a quartz Peltier cooled spray chamber and quartz torch) was used for all measurements. The combination of narrow ion energy distribution (due to ShieldTorch) and the 7900 ICP-MS's unique octopole-based cell enables efficient removal of interferences using an inert cell gas (Helium, He) and kinetic energy discrimination (KED). The use of He cell gas also eliminates safety issues related to reactive gases such as hydrogen, hydrogen mixes or ammonia.

The CRC technology ensures accurate results and also has a number of advantages over the reactive cell gases such as:

- He is an inert gas, so no new interferences are produced from analytes or from the matrix components during analysis
- He mode effectively removes all polyatomic interferences along with reactive polyatomics

The instrument operating conditions are summarized in Table 2.

Instrument Parameters	He mode
Plasma mode	General Purpose
RF power (W)	1550
Plasma gas flow (L/min)	15.0
Auxiliary gas flow (L/min)	0.9
Carrier Gas Flow (L/min)	1.03
Helium Cell gas flow (mL/min)	5.0
Detector mode	Auto
Integration time/mass (s)	0.20
Energy Discrimination (V)	5.0

Table 2. Agilent 7900 ICP-MS operating parameters.

Standard and sample preparation

The certified reference material SRM 1568b was analyzed during this work. 200 mg of SRM, noodle samples, flavor sachet and samples for spiking studies (1 µg/L, 2 µg/L and 3 µg/L concentration levels) were weighed in multiple replicates and digested in 2.5 mL nitric acid, 0.2 mL hydrogen peroxide and 0.1 mL hydrochloric acid using a microwave digestion device (program details are listed in Table 3) and made up to 10 mL using Milli-Q water. The method blank, spiked and SRM samples were prepared simultaneously. An internal standard, containing Ge, Rh and Bi, was added to all standards and samples at a concentration of 100 µg/L. All four elements, As, Pb, Cd and Hg, were analyzed using the Agilent 7900 ICP-MS in He mode in a single run. The schematic of the sample analysis workflow is depicted in Figure 1.

Step	Temperature (°C)	Ramp (min)	Hold time(min)
1	160	15	10
2	180	10	10
3	195	10	10

Table 3. Microwave Digestion Program.

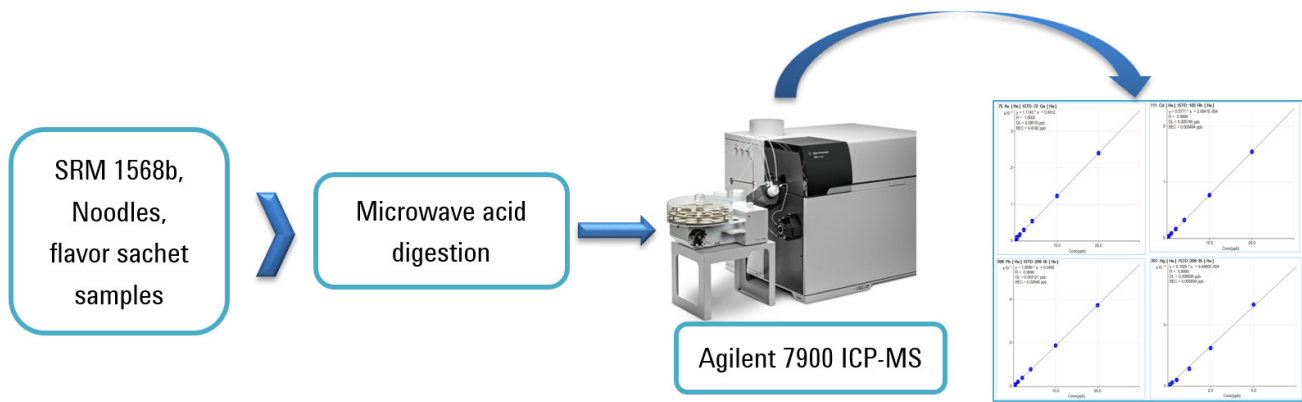


Figure 1. Schematic of sample analysis workflow using the Agilent 7900 ICP-MS.

RESULTS AND DISCUSSION

Calibration

In Table 4, the calibration coefficients, method detection limits and background equivalent concentrations are listed for the 4 elements and in Figure 2, the calibration curves from 10ppt up to 20ppb are shown. The system demonstrated good sensitivity, with detection limits at ng/L levels for all elements.

Element	R Value	Method detection limit, µg/L	Background Equivalent Concentration (BEC), µg/L	Method detection limit in sample, µg/kg
⁷⁵ As	0.9999	0.002911	0.008432	0.15
¹¹¹ Cd	0.9999	0.005746	0.003494	0.29
²⁰¹ Hg	0.9994	0.006581	0.005798	0.33
²⁰⁸ Pb†	0.9999	0.003121	0.02646	0.16

Table 4. Calibration coefficients, method detection limits and background equivalent concentration data. All elements were measured in He mode. †Pb data is based on the sum of the 208, 207 and 206 isotopes

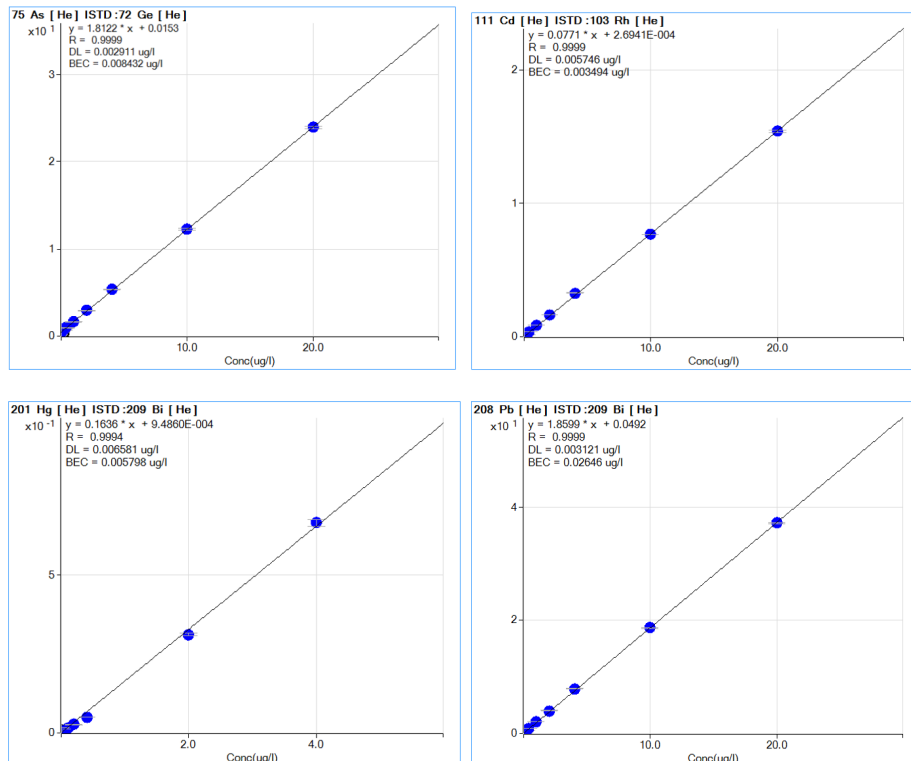


Figure 2. Calibration curves for As, Cd, Hg and Pb.

Method Validation

The Standard Reference Material (SRM) 1568b was analyzed for heavy metals, As, Cd, Hg and Pb, as an instrument check before starting the analysis of the commercial noodle, flavor sachet and spiked samples. The results in Table 5 show excellent agreement with the certified values for all selected elements. The internal standard, containing Ge, Rh and Bi, was added to all standards and samples at a concentration of 100 µg/L. The recovery and stability of these elements was monitored for 4 hours as shown in Figure 3.

SRM	Element	ISTD	Certified Value, mg/kg	Measured Value, mg/kg (n=6)	SD, %	Recovery, %
1568b	75 As [He]	Ge	0.285±0.014	0.2720	1.65	95.4
	111 Cd [He]	Rh	0.0224±0.0013	0.0239	0.68	104.6
	201 Hg [He]	Bi	0.00591±0.00036	0.00569	0.43	96.3
	208 Pb [He]	Bi	0.008±0.003	0.0086	0.14	107.2

Table 5. Recoveries for heavy metals in SRM 1568b.

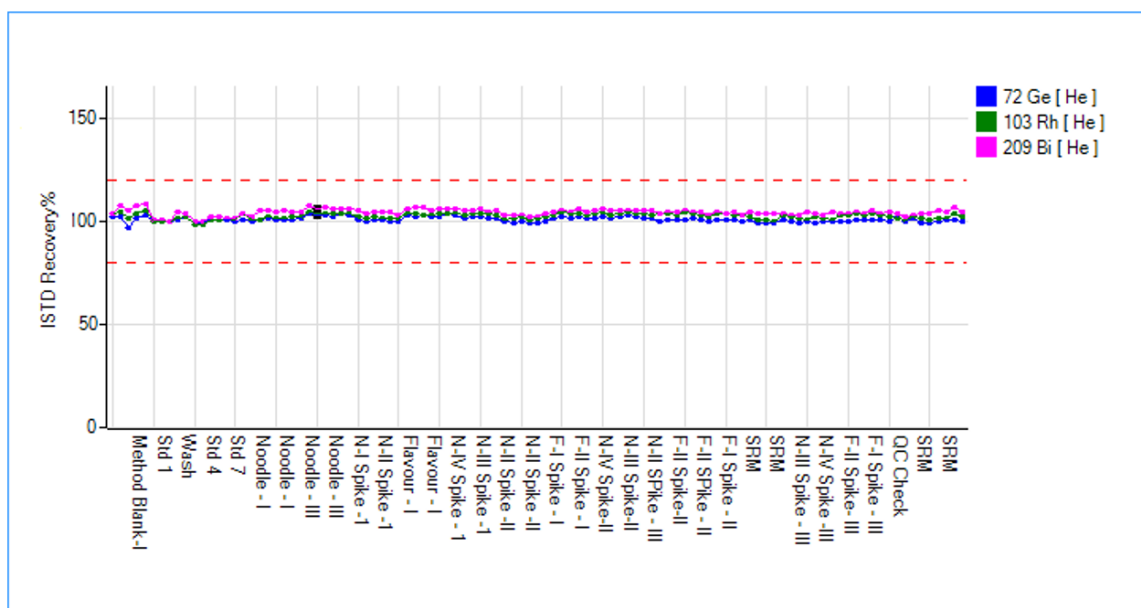


Figure 3. Internal Standard recovery and stability graph over 4 hours period.

Analysis of real samples and spiked samples

Along with the noodles samples, matrix spiked samples were also analyzed at 1 µg/L, 2 µg/L and 3 µg/L levels for all four heavy metals, with six replicates at each level. The recoveries obtained during these studies were between 90 – 110 %, with RSDs of less than 5%. The results are summarized in Tables 6 and 7.

Element	Noodles		Flavor Sachets
	Vendor A (n=6)	Vendor B (n=6)	Vendor C (n=6)
	Average, mg/kg	Average, mg/kg	Average, mg/kg
75 As [He]	ND*	ND*	ND*
111 Cd [He]	ND*	ND*	ND*
201 Hg [He]	ND*	ND*	ND*
208 Pb [He]	0.25	0.30	0.31

*ND: Less than the detection limit

Table 6. Real sample results for heavy metals.

Vendor	Element	ISTD	Noodles Spike Level - 1 (1 µg/L)		Spike Level – 2 (2 µg/L)		Spike Level – 3 (3 µg/L)	
			(n=6)					
			% Average Recovery	% RSD	% Average Recovery	% RSD	% Average Recovery	% RSD
Matrix : Noodles								
A	75 As [He]	Ge	100.2	3.49	98.5	1.8	95.3	1.72
	111 Cd [He]	Rh	104.0	1.69	103.4	1.9	102.6	0.72
	201 Hg [He]	Bi	95.5	3.77	100.6	2.89	105.2	1.41
	208 Pb [He]	Bi	102.8	3.99	104.7	1.99	104.5	1.1
B	75 As [He]	Ge	94.3	2.88	94.0	2.05	93.3	1.58
	111 Cd [He]	Rh	100.9	0.87	100.5	1.28	102.6	0.96
	201 Hg [He]	Bi	99.2	1.95	101.6	1.39	98.2	0.87
	208 Pb [He]	Bi	95.3	3.52	96.9	0.99	105.1	0.69
Matrix : Flavor Sachets								
C	75 As [He]	Ge	94.6	1.98	96.2	2.28	94.0	1.8
	111 Cd [He]	Rh	102.1	1.51	101.5	0.58	102.4	0.38
	201 Hg [He]	Bi	101.1	2.17	106.3	1.88	104.4	2.16
	208 Pb [He]	Bi	98.3	1.95	101.1	1.51	104.3	2.06

Table 7. Spiked sample results for heavy metals.

CONCLUSIONS

The Agilent 7900 ICP-MS operated in standard configuration offers a complete solution for the accurate determination of trace level heavy metals in noodles and flavor sachets, meeting the detection limits and recovery requirements of the Food Safety Standards Authority of India (FSSAI). The 7900 ICP-MS, with unique Helium mode capability, provides the utmost confidence, producing accurate and precise results. The Helium modes ability at reducing polyatomic interferences means there is no need for any reaction gas. This has been demonstrated on food products where no prior information was available on the sample matrix. Excellent results were obtained in terms of sensitivity, recovery and detection limits for As, Cd, Hg and Pb and good recovery results were achieved for SRM 1568b

REFERENCES

1. Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, Food Safety Standards Authority of India (FSSAI), Ministry of Health and Family Welfare, 2011.



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Printed in EU, 2016-03-14
5991-6766EN



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