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Application News

NO. SP-08-ADI-058

ICPMS-2030

Estimation of heavy metals in Infant food powder using Shimadzu inductively-coupled-plasma-mass spectrometry

Introduction

The human body needs small amounts of certain heavy metals, such as iron and zinc, to function properly. But cadmium, arsenic, lead, and mercury can be toxic for everyone and pose particular risks for young children.
Exposure to even small amounts of these heavy metals at an early age may increase the risk of several health problems, especially lower IQ and behavior problems, and have been linked to autism and attention deficit hyperactivity disorder.

Breast-feeding is the optimal mode of nutrition for infants. However, commercially available infant formulae provide a suitable alternative, especially when breast-feeding is not possible and/or not adequate.
Babies and toddlers are particularly vulnerable due to their developing brains and organ systems. The human body needs small amounts of certain heavy metals, such as iron and zinc, to function properly. Exposure to even small amounts of these heavy metals at an early age may increase the risk of several health problems, especially lower IQ and behavior problems, and have been linked to autism and attention deficit hyperactivity disorder.

Table 1: FSSAI permissible elemental Limits for Infant Food

Elements	FSSAI Limits (ppm)
Arsenic (As)	0.05
Cadmium (Cd)	0.1
Copper (Cu)	15
Mercury (Hg)	1.0
Lead (Pb)	0.02
Tin (Sn)	5
Zinc (Zn)	50

Experimental

A Sample of Infant food was purchased locally for the extraction of metal elements for this study. Based on FSSAI limits[1][2] (Table 1), limit of quantification (LOQ) were set at 20% of maximum residual limit (MRL). Concentrations mentioned are as per below Table 2.

Table 2: LOQ limits set for experimentation

Elements	LOQ (ppm)	10x LOQ (ppm)
Arsenic (As)	0.01	0.10
Cadmium (Cd)	0.02	0.20
Copper (Cu)	3	30
Mercury (Hg)	0.2	2.0
Lead (Pb)	0.004	0.04
Tin (Sn)	1	10
Zinc (Zn)	10	100

Sample Preparation:

0.5g of Infant food sample powder was accurately weighed into a microwave vessels. Samples were kept for pre digestion after carefully adding ultra pure water, nitric acid (HNO_3) and hydrochloric acid (HCl). The vessels were then heated in microwave digestion system (MDS) under controlled temperature program (Table 3).

Pre spiked recovery studies were carried out at LOQ, & 10x LOQ levels (considering weight & dilution factor) by spiking samples with standard solution of metals and digested under similar circumstances as that of sample.

After digestion, samples were left to cool to ambient temperature and filled up using ultra-pure water in a standard volumetric flask (25ml).

Table 3: Microwave digester programme

Steps	Ramp (min)	Temp (°C)	Hold time (min)
1	10	120	05
2	10	180	20

The digested solutions obtained are analysed on Shimadzu ICPMS-2030. Yttrium (Y) and bismuth (Bi) were added as internal standards (IS). Gold solution and IS solution are added at a final concentration of 100ppb and 10 ppb respectively to blank, samples and linearity standards.
Note: Gold (Au) is added to stabilize Hg

Calibration standard preparation

Sigma Aldrich 1000 ppm individual certified reference standards were used for preparation of intermediate stock solution. Calibration linearity solutions were prepared by diluting intermediate stock solution as shown in Table 4.

Analytical Conditions

A Shimadzu ICPMS-2030 coupled with auto sampler AS-10 (Figure 1) was used. The detailed instrument configurations and operating parameters are summarized in Table 5.

Figure 1. ICPMS-2030 Inductively coupled plasma mass spectrometer with AS-10.

Table 5: Instrumental parameters

Plasma Torch	Mini torch
Radiofrequency	1.2 kW
Sampling depth	5 mm
Plasma gas flow rate	10 L/min
Auxiliary gas flow rate	1.1 L/min
Carrier gas flow rate	0.7 L/min
Collision gas	Helium
Collision gas flow rate	6.0 mL/min
Chamber	Cyclone chamber (electronically cooled)
Chamber temperature	5° C

Results

1. Toxic elements such as As, Cd, Hg and Pb found to be at much lower concentration in sample.
2. The Calibration standard showed good linear response (Figure 2) with correlation coefficient ≥ 0.997 for all elements.
3. % RSD for each aspiration throughout the sequence were less than 10% (comprises blank, standard and sample).
4. %RSD of result obtained for 4 preparation (n=4) are less than 6%.
5. Spike recoveries for all samples were between ±20%.

Table 4: Calibration standard solution concentration (ppb)

Element	As	Cd	Cu	Hg	Pb	Sn	Zn
Calibration Std Level 1	0.1	0.2	30	2	0.04	10	100
Calibration Std Level 2	0.2	0.4	60	4	0.08	20	200
Calibration Std Level 3	0.3	0.6	90	6	0.12	30	300
Calibration Std Level 4	0.5	1	150	10	0.2	50	500
Calibration Std Level 5	1	2	300	20	0.4	100	1000
Calibration Std Level 6	2	4	600	40	0.8	200	2000
Calibration Std Level 7	2.5	5	750	50	1	250	2500

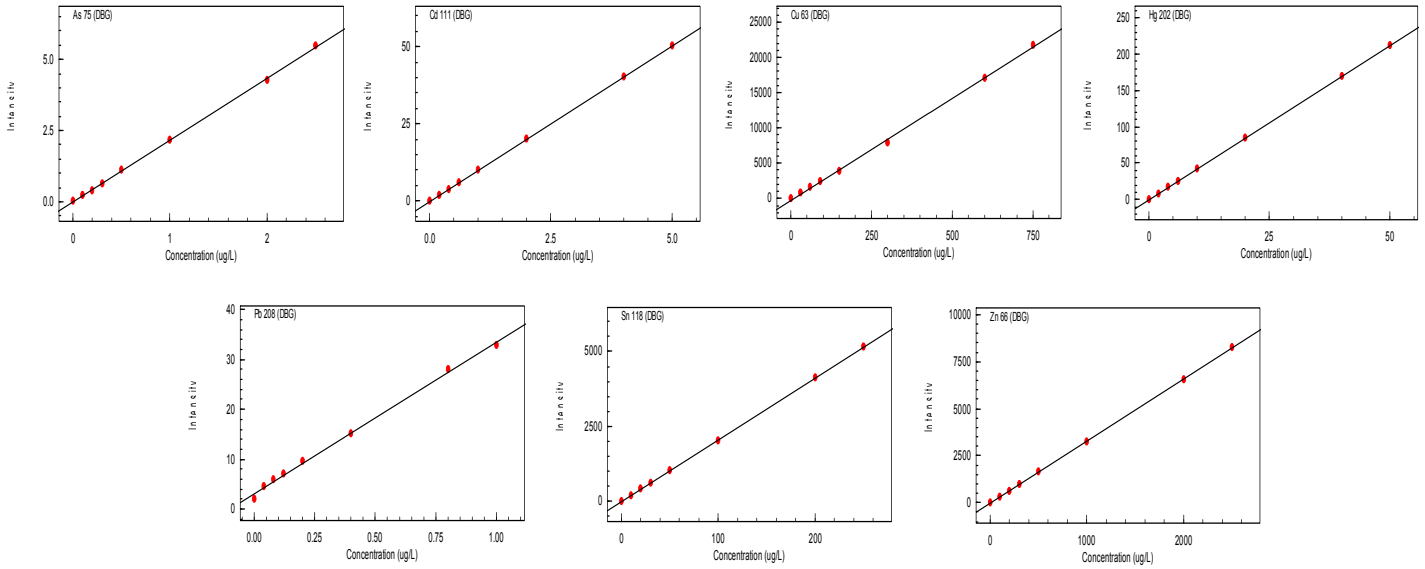

Figure 2. Linearity curves for targeted elements

The results obtained were evaluated for statistical parameters like accuracy, precision and linearity. Accuracy in terms of recovery was found to be between 80 to 120% for pre-spiked samples. Precision in terms of %RSD is found to be less than 6% for all spike samples. Accuracy and % RSD for precision for Infant food are shown in Table 6.

Table 6: Average accuracy at LOQ, 10x LOQ (n=4 replicates)

Elements	LOQ spike %recovery	LOQ %RSD	10x LOQ spike %recovery	10x LOQ %RSD	Content in Sample (ppm)
As	119.8	5.50	113.6	2.04	0.012
Cd	92.9	2.45	95.6	1.62	BLOQ
Cu	79.8	5.82	92.8	0.51	3.67
Hg	101.0	1.79	103.2	1.07	BLOQ
Pb	100.6	4.38	101.8	0.83	0.03
Sn	93.5	0.84	96.7	1.02	BLOQ
Zn	82.8	1.51	98.1	1.71	36.83

Note: BLOQ = Below Limit Of Quantitation

Conclusion

Trace metals in Infant food sample were analysed following limits as per FSSAI guideline using ICPMS-2030. High sensitive, selective & accurate ICPMS method was developed for Infant food. Thus, ICPMS is proven to be best tool for metal analysis of food matrices.

References

[1] Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, 2011
[2] AOAC Official Method 2015.01

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