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Application News

NO. SP-010-ADI-066

ICPMS-2030

Trace elemental analysis of chicken egg using Shimadzu Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Introduction

Egg is an important source of high quality natural proteins, lipids, fatty acids, vitamins and essential minerals. Chemical composition of egg is complicated due to presence of many organic compounds that can bind trace elements e.g. protein ovalbumin attracts Se, Hg, Zn, Cu, Mn, glycoprotein ovotransferrin and phosphoproteins bind Fe ions.
The elemental content of egg need to be checked for food safety assessment. It should be considered that essential elements may become toxic if consumed in excessive amount or contrary, may lead to element deficiency if consumed insufficiently.
Poultry feed composition is different in various parts of the world. It reflects in elemental content of egg yolk and albumen. The contamination of elements in egg can be because of microorganism and by chemical substances which are transferred from environment into food chain. The aim of the present study was to quantitate trace elements in chicken egg in accordance with FSSAI[1,2] Maximum Residual Limits (MRLs) as shown in Table 1.

Experimental

Chicken egg samples were purchased from local market. The edible part of egg was vortexed to homogenise the content.

Table 1: FSSAI MRLs (ppm) for egg

Elements	FSSAI MRLs
Arsenic (As)	1.1
Cadmium (Cd)	1.5
Copper (Cu)	30
Mercury (Hg)	1
Lead (Pb)	0.1
Selenium (Se)*	2.5
Tin (Sn)	250
Zinc (Zn	50

***The limit for Se is not available as per FSSAI, so value was taken arbitrarily.**

Sample Preparation

About 500 mg of sample was weighed and transferred into microwave vessels. Samples were kept for pre digestion.
After carefully adding 5 mL of suprapure nitric acid, 0.5 mL suprapure hydrogen peroxide and 1 mL ultrapure water. The vessels were then heated in microwave digestor system under controlled temperature program (Table 2).
After digestion, samples were cooled to ambient temperature and transferred to a 50 mL volumetric flask and diluted with ultrapure water. Pre-spiked recovery samples were prepared at LOQ & 10 x LOQ levels.

Table 2: Microwave digestion program

Ramp (min)	Temp (°C)	Hold time (min)
10	120	05
10	180	30

Calibration standard preparation

Calibration standards were prepared form 1000 ppm certified reference standards after appropriate dilutions, covering concentration range from 10 to 250 % of the MRL. The LOQ was set to 20 % of the MRLs in accordance with commission regulation (EU) 836/2011 [3] (Table 3).

Table 3: LOQs (ppm) for egg in present work

Elements	LOQs
Arsenic (As)	0.22
Cadmium (Cd)	0.3
Copper (Cu)	6
Mercury (Hg)	0.2
Lead (Pb)	0.02
Selenium (Se)	0.5
Tin (Sn)	50
Zinc (Zn)	10

Analytical Conditions

A Shimadzu ICPMS-2030 coupled with auto sampler AS-10 (Figure 1) was used for analysis of measuring elements in egg. The detailed instrument configuration and operating parameters are summarized in Table 4.
The elements Bi, Ge and Y were used as internal standards.

Continuous Calibration Verification (CCV) checks were run in between to check drift of the system throughout the run. The standard of LOQ concertation was run as CCV. The concentrations of linearity standards are shown in Table 5.

Table 4: Instrumental parameters

Plasma torch	Mini torch((P/N:S211-94018))
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
Chamber temperature	5 °C

Figure 1. Shimadzu ICPMS-2030 with Autosampler AS-10

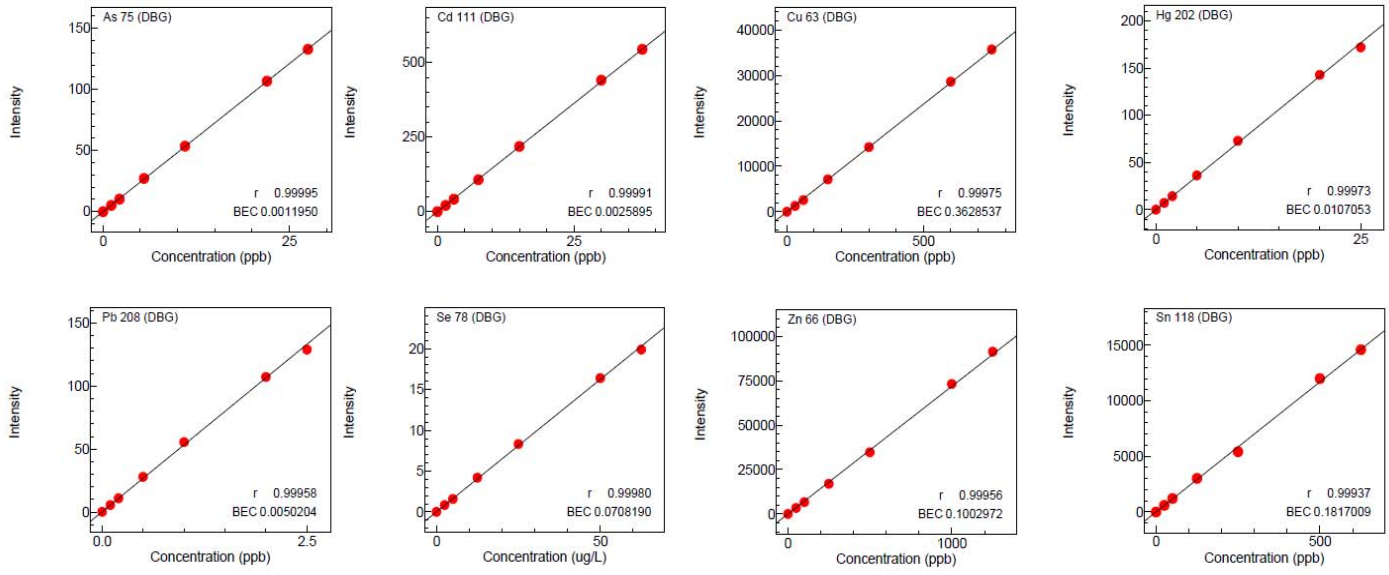

Fig 2. Calibration curves obtained in present study

Results

The calibration standards showed good linear response with correlation coefficient (r) ≥ 0.999 for all elements. The content for all analyzed elements was lower than the set LOQ. The % recoveries obtained are given in Table 6. The recoveries at LOQ and 10 x LOQ levels were between 80 to 120 % for all elements.

% RSD of result obtained for 4 preparation are less than 8 %, indicating good repeatability. Results of CCVs are shown in Table 7.
Accuracy of CCVs was between 90 to 110 %.

Table 5: Calibration of linearity standards in ppb prepared in present study

Elements	As	Cd	Cu	Hg	Pb	Se	Sn	Zn
Calibration Std Level 1	0	0	0	0	0	0	0	0
Calibration Std Level 2	1.1	1.5	30	1	0.1	2.5	25	50
Calibration Std Level 3	2.2	3	60	2	0.2	5	50	100
Calibration Std Level 4	5.5	7.5	150	5	0.5	12.5	125	250
Calibration Std Level 5	11	15	300	10	1	25	250	500
Calibration Std Level 6	22	30	600	20	2	50	500	1000
Calibration Std Level 7	27.5	37.5	750	25	2.5	62.5	625	1250

Table 6: Average % recovery and % RSD at LOQ, 10 x LOQ (n=4 replicates)

Elements	LOQ % recovery	LOQ % RSD	10 x LOQ % recovery	10 x LOQ % RSD
As	102.9	2.7	104.6	0.7
Cd	97.5	1.4	103.2	0.7
Cu	89.8	1.6	104.5	1.1
Hg	99.6	4.2	98.3	3.8
Pb	109.5	6.4	100.8	4.1
Se	110.6	4.0	109.4	0.9
Sn	101.2	4.1	104.8	1.9
Zn	88.5	4.7	99.7	0.6

Table 7: Results for % accuracy of the CCVs

CCV standard	As	Cd	Hg	Pb	Cu	Se	Sn	Zn
CCV 1	97.3	99.3	90.3	104.5	102.5	96.6	99.2	92.8
CCV 2	96.8	96.7	90.8	98.5	103.5	96.8	101.6	92.0
CCV 3	94.1	95.3	91.5	100.0	103.5	95.8	101.4	92.3

Conclusion

Present work demonstrates a robust and reliable digestion method for determination of trace elements in egg by ICPMS 2030. The excellent spike recoveries obtained shows efficacy of the digestion method for a complex matrix like egg. The % recovery and % RSD obtained shows the reproducibility of the method.

References

[1] Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, 2011

[2] Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, 2006

[3] Commission regulation, EU No 836/2011

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Shimadzu Analytical (India) Pvt.Ltd.
1 A/B, Rushabh Chambers, Makwana Road,
Marol, Andheri (East), Mumbai- 400059, India.
Tel: +91 22 29204741 Fax: +91 22 29205679
www.shimadzu.in