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Application News

NO. SP-05-ADI-055

ICPMS-2030

Estimation of heavy metals at trace level in refined sugar using inductively-coupled-plasma-mass spectrometry

Introduction

White refined sugar is crystallized sucrose of a high purity that is industrially produced from sugar beet or sugar cane. It commonly used for baking, cooking, sweetening domestic products and beverages or in preparation of processed food etc. It may contain some soluble and /or insoluble organic and inorganic impurities that affect its quality and market value. Inorganic impurities may come from plat cultivation place, raw material, chemicals, manufacturing- refining process etc.

Presence of trace level elements like Arsenic (As), Cadmium (Cd), Chromium (Cr), Copper (Cu), Mercury (Hg), Lead (Pb) and Tin (Sn) in sugar cause adverse effects on human health. Thus, regulatory bodies like Food Safety and Standards Authority of India (FSSAI) & Food and Drug Administration (FDA) has recommended safety limits (Table 1) for trace metal contaminants in sugar.

At concentrations of sub parts per-billion level in solution, Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a vital tool for providing sensitive, selective, accurate, fast, reliable tool to determine metals in sugar at trace level.

Table 1: FSSAI permissible elemental Limits for Sugar

Elements	FSSAI Limits (ppm)
Arsenic (As)	1.0
Cadmium (Cd)	1.5
Chromium (Cr)	0.02
Copper (Cu)	20
Mercury (Hg)	1.0
Lead (Pb)	5.0

Experimental

A white refined sugar sample was selected for the extraction of trace metals for this study. Based on FSSAI limits[1][2] , limit of quantification (LOQ) were set (Table 2).

Table 2: LOQ limits set for experimentation

Elements	LOQ (ppm)	2x LOQ (ppm)	4x LOQ (ppm)
Arsenic (As)	0.04	0.08	0.16
Cadmium (Cd)	0.04	0.08	0.16
Chromium (Cr)	0.01	0.02	0.04
Copper (Cu)	0.04	0.08	0.16
Mercury (Hg)	0.04	0.08	0.16
Lead (Pb)	0.04	0.08	0.16
Tin (Sn)	0.04	0.08	0.16

Sample Preparation:

Samples were prepared by accurately weighing 0.5 g into a microwave vessels. Samples were kept for pre digestion after carefully adding of ultra pure water, nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). The vessels were then heated in microwave digestion system (MDS) under controlled temperature per programme (Table 3).

Pre spiked recovery studies were established at LOQ, 2x LOQ & 4x LOQ levels (considering weight & dilution factor) by spiking sugar samples with standard solution of elements and digested under similar conditions as that of sample.

After digestion, samples were cooled to ambient temperature and made up (20mL) using ultra-pure water.

Table 3: Microwave digester programme

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

The extracts obtained were analysed on Shimadzu ICPMS-2030. Scandium (Sc), Yttrium (Y) and Bismuth (Bi) were added as internal standards at a final concentration of 10 ppb to all samples and linearity standards.

Calibration standard preparation

Sigma Aldrich 1000 ppm individual certified reference standards were used for preparation intermediate stock solution. Calibration standard solutions were prepared by diluting intermediate stock solution. 0.4 ppb to 20 ppb linearity standards were prepared for As, Cd, Cu, Hg, Pb, Sn and 0.1 ppb to 5.0 ppb for Cr in matrix matched acid.

Well known “Memory effect” for mercury was minimized through addition of 100 ppb gold solution in all standards and sample solutions to preserve mercury by amalgamation which prevents mercury volatilization and adsorption losses.

Analytical Conditions

A Shimadzu ICPMS-2030 coupled with auto sampler AS-10 (Figure 1) was used for target element analysis from sugar.
Measurements were made using an off axis collision cell system with helium gas which achieves superior sensitivity by providing molecular ion removal and high elemental ion transmission which in result reduces matrix interferences. The detailed instrument configuration and operating parameters are summarized in Table 4.

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

Table 4: Microwave digester programme

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	6.0mL/min
Chamber	Cyclone chamber (electronically cooled)
Chamber temperature	5°C

Results

Quantification of trace metals (As, Cd, Cr, Cu, Hg, Pb, Sn) was performed using internal standard method. The internal standard elements were selected for target elements according to the best possible match in terms of proximity with respect to mass to charge ratio and ionization behavior.

LabSolutions ICPMS software guide in selection of best elemental masses based on their isotopic ratio, background equivalent concentration (BEC) values and isobaric interference.

1) Extracts were analysed on ICPMS-2030 using LabSolutions QuanBase mode. Quantitative results, 3s (LOD) & 10s (LOQ) values reported in Table 5.
2) Accuracy in terms of recovery was evaluated for all spiked samples. Recoveries were found to be between 80 to 120% for all elements shown in Table 5.
3) Good linear response obtained for calibration standard solutions with correlation coefficient ≥ 0.999 for all elements (Figure 2).
4) Average %RSD obtained for all standard and samples (n=6) were less than 5%. This shows good precision of the method.

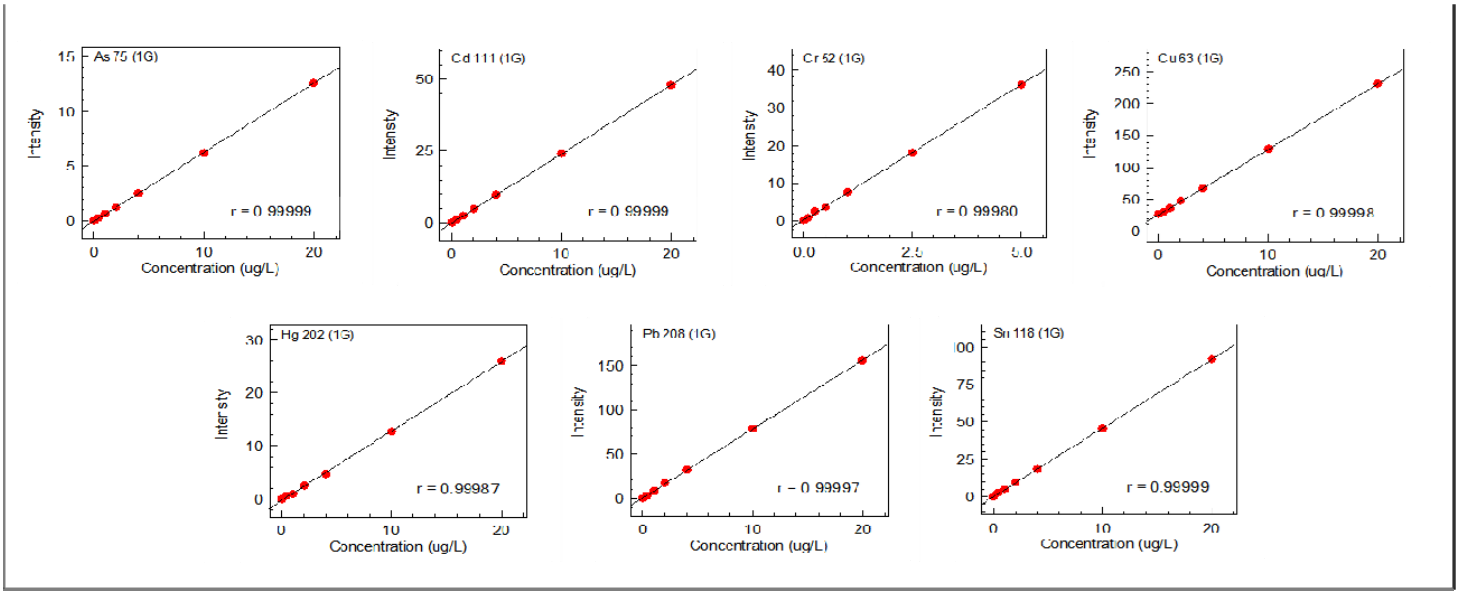

Figure 2. Linearity curves for targeted elements

Table 5: Concentration of elements, LOD & LOQ, Average accuracy at LOQ, 2x LOQ, 4x LOQ level (n=6 replicates)

Elements	Sugar (ppb)	3s (ppb)	10s (ppb)	LOQ spike recovery	2x LOQ spike recovery	4x LOQ spike recovery
As	BLOQ	0.0068	0.0227	109.1	110.8	99.4
Cd	BLOQ	0.0014	0.0047	98.6	96.1	86.5
Cr	BLOQ	0.0006	0.0018	106.6	94.2	98.8
Cu	BLOQ	0.1156	0.3852	99.5	89.8	85.3
Hg	BLOQ	0.0059	0.0196	94.3	98.7	87.3
Pb	BLOQ	0.0016	0.0052	106.0	106.2	102.8
Sn	BLOQ	0.0049	0.0162	101.5	93.4	90.5

Note: BLOQ = Below Limit Of Quantitation

Conclusion

These experimental results confirm the effectiveness and the capability of Shimadzu ICPMS-2030 to analyze trace level elements in sugar samples. Recovery studies proved validation of microwave digestion sample preparation method. New octaplate collision cell technology of ICPMS-2030 offers high throughput and great sensitivity by removing polyatomic interferences. LabSolutions ICPMS software function simplifies the process of developing analytical methods.

ICPMS-2030 is best tool for elemental quantification at trace level in sugar and other related food matrices.

References

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

First Edition: Sep. 2018

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