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Analysis of toxic elements in processed milk products using ICP-MS

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Introduction

Milk is considered as complete food as it provides all essential macro and micronutrients. Macronutrients consist of protein, carbohydrate and lipids, whereas micronutrients consist of elements, vitamins and enzymes[1][2]. Milk as an excretion of the mammary gland can carry numerous xenobiotic substances that constitute a technological risk factor for milk and milk products.
Toxic elements like Arsenic (As), Cadmium (Cd), Lead (Pb) and Mercury (Hg) have caused adverse effects on human health. These can be transferred from contaminated soil to plants and grass, causing accumulation of these toxic metals in cattle, but also in humans consuming milk. Milk processing may also cause contamination of milk products with toxic elements.
The objective of this study is to develop a sensitive, selective, accurate and reliable method using Shimadzu ICPMS-2030 to determine the risk of toxic heavy metals in milk product.

Figure 1 Milk and processed milk products

Methods and Materials

Commercially available milk products e.g. toned milk and cheese (Figure 1) were used for the extraction of toxic heavy metals in this study. Recovery studies were established by spiking milk product samples with standard solution of elements. The extracts obtained were analysed on Shimadzu ICPMS-2030.

Sample Preparation:

The samples were digested using microwave digestion system (Anton Paar).

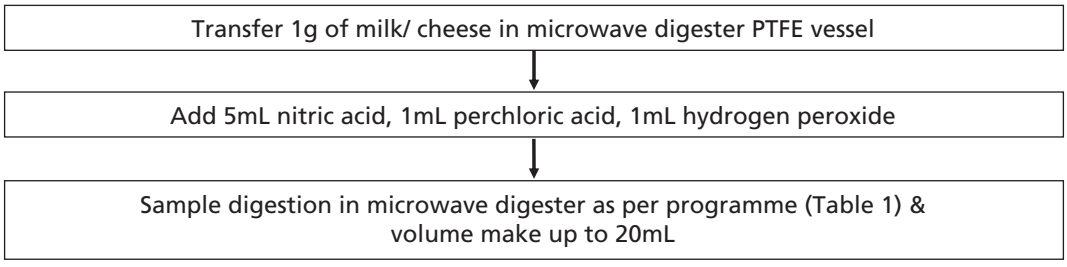

Table 1: Microwave digester programme

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

The extracts obtained were analysed on Shimadzu ICPMS-2030. Analysis was done using plasma generated by specially designed mini torch as an ionization source. ICPMS LabSolutions software and special features like Profile integration time and Total mass measurement were used for identification, detection and quantitation. NIST traceable standards were used for quantification of elements at low level in QuantBase mode (used for quantitation purpose).

Key features of ICPMS-2030

Inductively coupled plasma mass spectrometer ICPMS-2030 by Shimadzu (shown in Figure 3), sets a new benchmark in ICP-MS technology with minimum consumption of Argon gas. This system ensures highest quality of data, with very high degree of reliability. The newly developed collision cell (shown in Figure 3) based on UFsweeper technology uses high purity Helium gas for removal of polyatomic interferences. The mini-torch design allows the operation of ICPMS at low RF power without compromising the sensitivity.

Figure 2 Shimadzu ICPMS-2030 Inductively coupled plasma mass spectrometer

Figure 3 Newly developed Collision Cell

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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

Linearity data for elemental standards in the concentration range of 0.1 µg/L to 10 µg/L for toned milk and cheese is shown in Figure 4 and Figure 5.

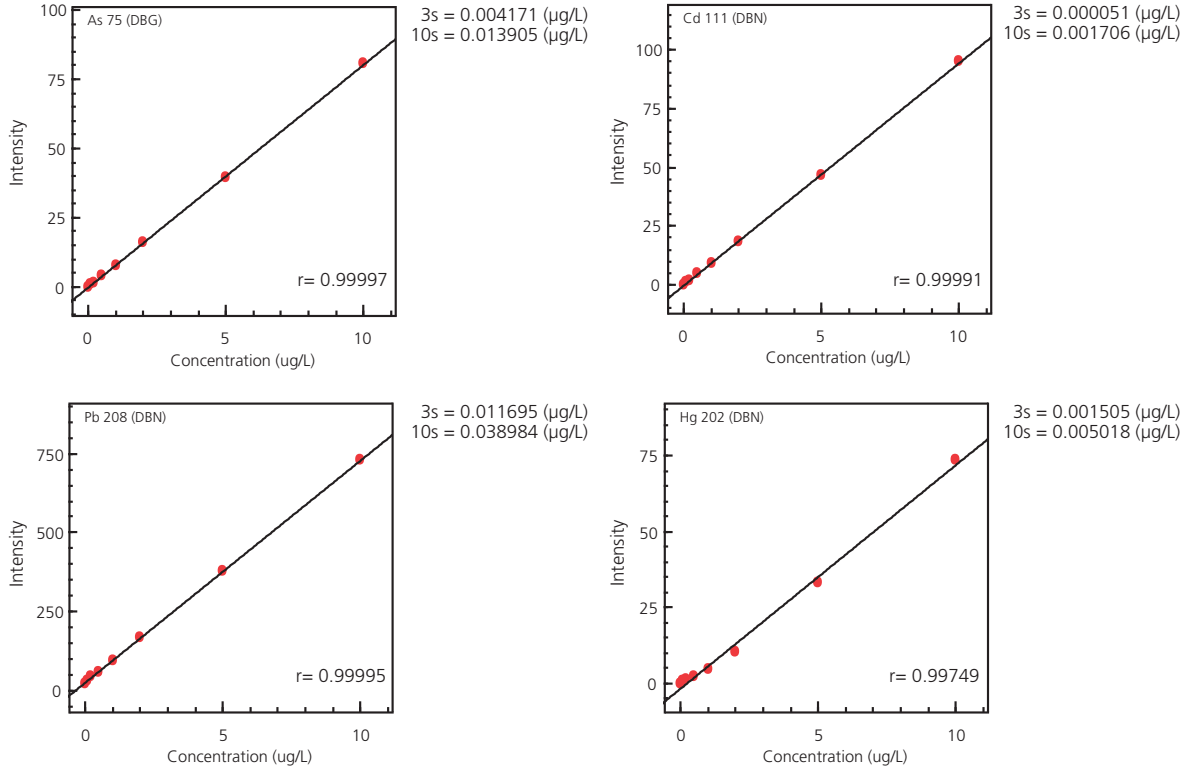

Figure 4 Standard linearity curves for 0.1-10µg/L for Milk

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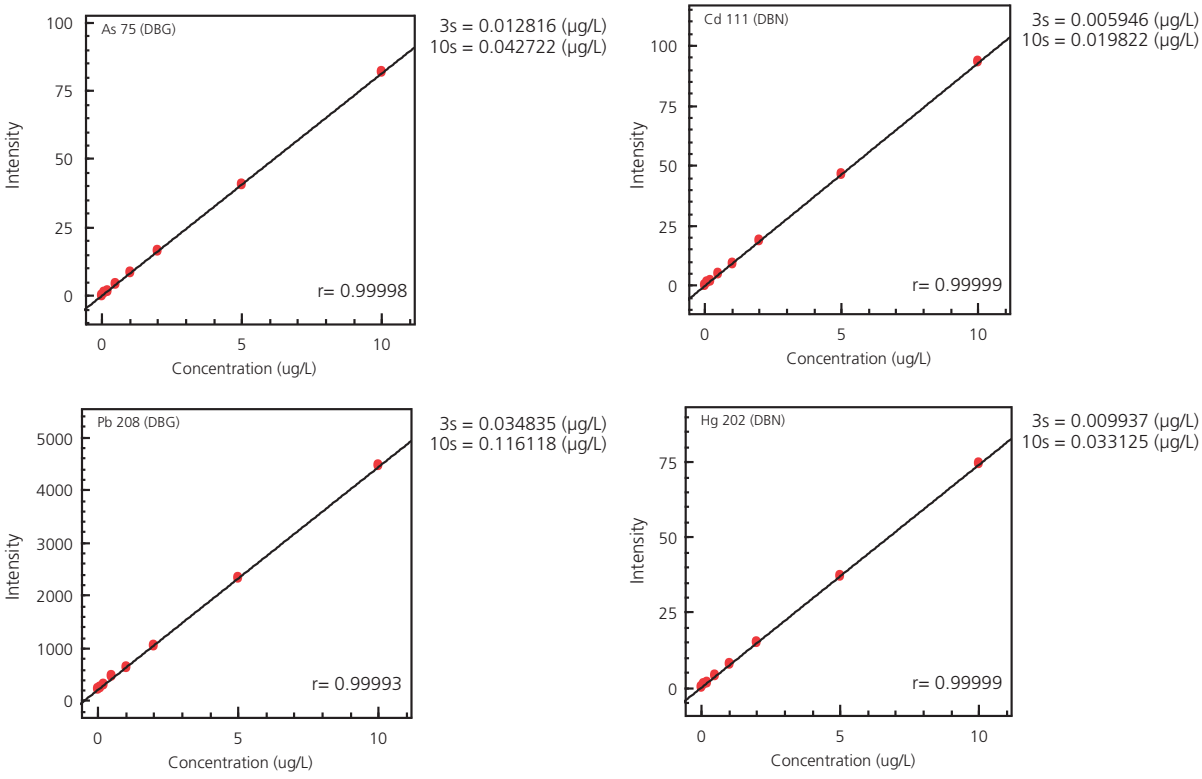

Figure 5 Standard linearity curves for 0.1-10µg/L for Cheese

Results

Extracts were analysed on ICPMS-2030 in QuanBase mode. The elements were further quantified using calibration curves plotted from analytical standards (Merck). Measurements were made using an off axis Collison cell system which helped to remove neutrals and photon interferences.

The results obtained were evaluated for statistical parameters like accuracy and linearity. Accuracy in terms of recovery was found to be between 70 to 120% for pre-spiked samples. The results showed good linear response with correlation coefficient ≥ 0.995.. The results obtained for toned milk and cheese are given in Table 1. RSD and % recoveries for toned milk and cheese are shown in Table 2 and Table 3 respectively. The RSD of six replicates was within 7%, showing good precision of the method.

Table 2 Average elemental results obtained for processed milk products (n = 6 replicates)

Elements	Toned milk (µg/L)	Cheese (µg/L)
As	Not detected	5.0
Cd	Not detected	Not detected
Pb	5.6	Not detected
Hg	1.6	1.2

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Table 3 Average accuracy results at 0.25 µg/L of for toned milk & cheese sample (n = 6 replicates)

Elements	Toned milk		Cheese	
	%Recovery (Accuracy)	%RSD	%Recovery (Accuracy)	%RSD
As	95-108	5.4	96-117	6.8
Cd	87-98	1.6	92-97	2.2
Pb	75-106	2.1	99 -115	6.3
Hg	100-110	0.9	101-107	2.4

Conclusion

ICPMS-2030 was found to be best technique for the determination of toxic heavy metals at very low concentration. The above methodology proved to be selective, sensitive, accurate and reliable.

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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