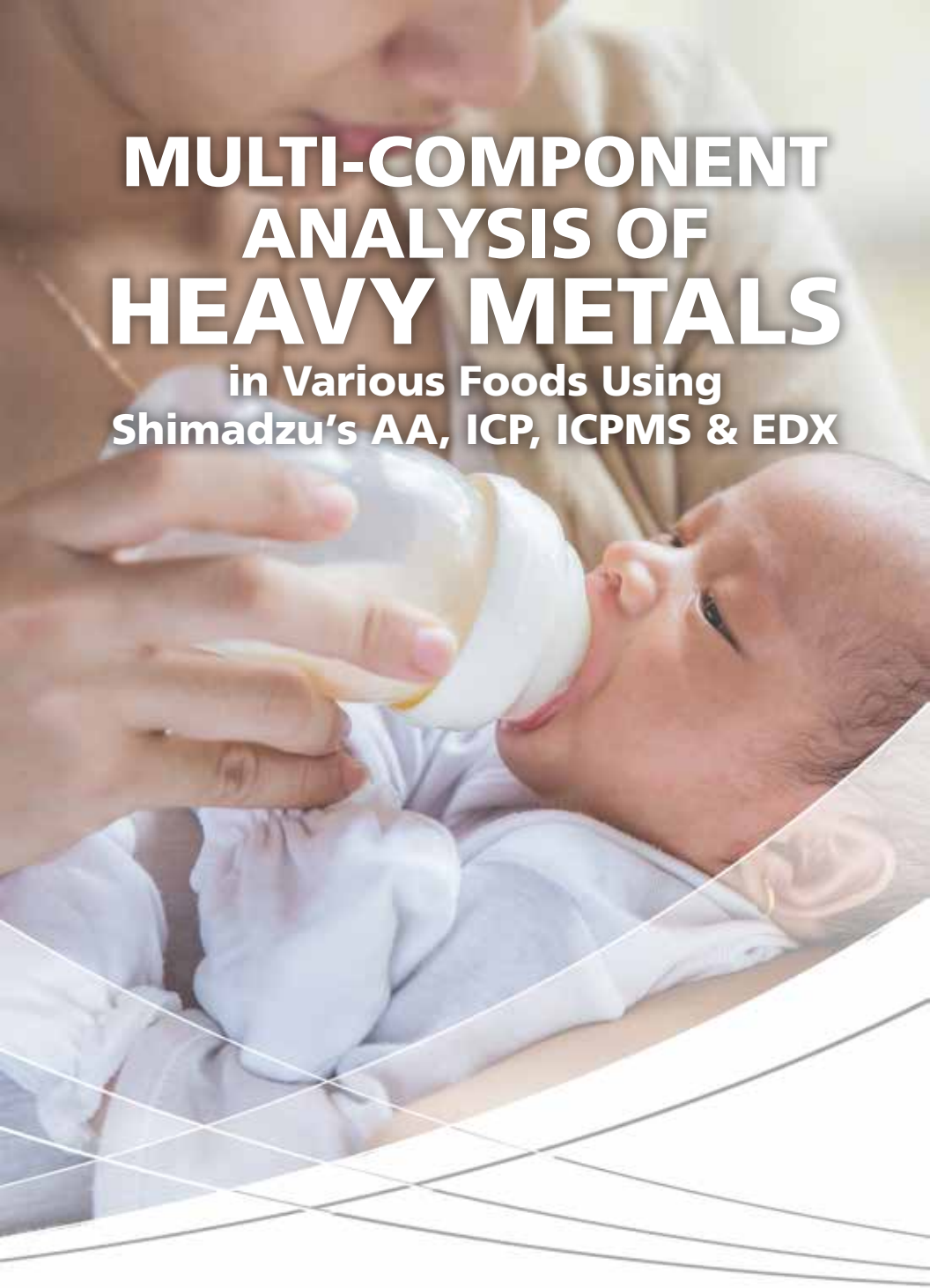


MULTI-COMPONENT ANALYSIS OF HEAVY METALS

in Various Foods Using Shimadzu's AA, ICP, ICPMS & EDX

SHIMADZU

Excellence in Science

HEAVY METALS TESTING

HEAVY METALS POSE A SIGNIFICANT THREAT TO HUMAN HEALTH

The U.S. Food and Drug Administration (FDA) identifies chemical hazards as serious threats to the food supply. Heavy metal contamination is among the many chemical hazards included in the official FDA guidance documents. Chemical contamination can be introduced at any stage in food production and processing. Heavy metals (e.g., cadmium, arsenic and mercury) can contaminate foods through agricultural practices or the use of contaminated soil or water. Additionally, heavy metals from equipment, containers or utensils can leach into food during processing and production.

Human consumption of heavy metals can result in serious health conditions. Lead exposure can impair cognitive development in children. Inorganic arsenic has been associated with cancer, skin lesions, developmental effects, cardiovascular disease, neurotoxicity and diabetes. To prevent exposure to toxic metals, testing laboratories need analytical instrumentation of the highest quality.

DETECTING TOXIC METALS IN FOOD PRODUCTS

Shimadzu's analytical instruments enable detection of toxic metals in food at even the lowest concentration levels. Atomic absorption (AA) spectrophotometers, inductively coupled plasma (ICP) optical emission spectrometers, and inductively coupled plasma mass spectrometers (ICPMS) offer high-sensitivity analysis of trace elements in foods. X-ray fluorescence spectrometers can perform elemental analysis without sample pretreatment. These instruments are ideal for both screening and emergency analysis. They enable simultaneous detection of trace and major elements in all types of food.

Simultaneous analysis of 19 elements in infant formula

SIMULTANEOUS DETERMINATION OF VARIOUS ELEMENTS IN INFANT FORMULA

Infant formula elemental analysis can be difficult because of the different concentration levels of each targeted element. To overcome this challenge, we developed a fast, easy-to-use method for simultaneous analysis of 19 elements in infant formula using the Shimadzu ICPMS-2030.

Equipped with a channeltron detector, the ICPMS-2030 combines high sensitivity (trace detection) with a wide dynamic range (10^9), which is key for simultaneous determination of major and trace elements. We measured infant formula samples in triplicate and spiked one of them with known concentration values to test method accuracy. An internal standard solution in 6% nitric acid was automatically mixed online with the sample before it was aspirated. Our LabSolutions software automatically chose internal standards (ISTD) and concentrations for each studied element.

Results for trace elements and contaminants in the two infant formulas are shown in the tables below. The quantitation results demonstrate that Shimadzu's ICPMS-2030 simultaneously and accurately quantifies the various elements present in infant formula samples.

Element	^{43}Ca*	^{53}Cr**	^{63}Cu**	^{56}Fe*	^{39}K*	^{25}Mg*
INF1	4282	50	3207	43	5796	548
INF2	7090	75	2918	72	8972	627
Element	^{55}Mn**	^{95}Mo**	^{23}Na*	^{31}P*	^{78}Se**	^{66}Zn*
INF1	655	93	1653	2728	316	56
INF2	611	175	2908	4446	275	52

Table 1: Trace elements concentration in studied infant formula (*ppm; **ppb)

Element (ppb)	^{27}Al	^{75}As	^{133}Cs	^{198}Hg	^{206}Pd	^{171}Sb	^{118}Sn
INF1	894	4.1	9.7	2.7	5.9	3.0	18.9
INF2	1249	5.4	21.2	2.3	3.1	2.4	18.2

Table 2: Contaminants concentration in studied infant formula

The quantitation results demonstrate that Shimadzu's ICPMS-2030 simultaneously and accurately quantifies the various elements present in infant formula samples.

ANALYSIS OF TOXIC ELEMENTS IN PROCESSED MILK PRODUCTS

Milk is considered a complete food because it provides essential macronutrients (protein, carbohydrates and lipids) and micronutrients (elements, vitamins and enzymes). However, because milk is excreted by the mammary gland, it can carry numerous xenobiotic substances that can contaminate milk and milk products.

Toxic elements like arsenic (As), cadmium (Cd), lead (Pb) and mercury (Hg) can have adverse effects on human health. These toxins can be transferred from contaminated soil to plants and grass to cattle to the milk they produce—and then to humans who consume that milk. Milk processing methods may also cause contamination.

Using the Shimadzu ICPMS-2030, we developed a sensitive, selective, accurate and reliable method to detect toxic heavy metals in milk products. To evaluate our method, we analyzed commercially available toned milk (buffalo milk with added skim milk, powdered skim milk and water) and cheese for toxic heavy metals. Then, we spiked the milk product samples with a standard solution of the toxic elements.

Shimadzu's ICPMS-2030 proved to be the most accurate and reliable instrument for determining toxic heavy metals at very low concentrations.

Element	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

Table 3: Average elemental results obtained for processed milk products (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

Table 4: Average accuracy results at 0.25 µg/L of for toned milk and cheese sample (n=6 replicates)

We analyzed the extracts with plasma generated by a specially designed mini-torch. All ICPMS-2030 instruments include our LabSolutions software for fast, reliable analysis of multi-analyte data. Special features like profile integration time and total mass measurement were used for identification, detection and quantitation.

Recovery accuracy was between 75% and 115% for pre-spiked samples. The results showed good linear response with a correlation coefficient generally ≥ 0.9999 except for mercury in milk. The relative standard deviations (RSDs) and percentage recoveries for the toned milk and cheese samples are shown in the table above. The RSD of six replicates was within 7%, demonstrating the precision of the method. Shimadzu's ICPMS-2030 proved to be the most accurate and reliable instrument for determining toxic heavy metals at very low concentrations.

QUANTITATIVE ANALYSIS OF CHROMIUM IN FOOD PACKAGING

Speciation analysis is the process of separating and quantifying one or more individual species in a sample. It is critical for determining the presence of elements like cadmium, chromium, mercury, tin and arsenic in food samples. For example, this process is essential when testing food packaging for chromium. Analysts must determine the presence of the hexavalent chromium species because it is recognized as a human carcinogen.

Using the Shimadzu ICPMS-2030, connected to our Prominence Inert HPLC System, we conducted chromium speciation for food packaging analysis. A basic mini-torch setup offered drastically lower flow rates of argon. We obtained excellent sensitivity within a low ppt concentration range as well as a good calibration curve linearity for both Cr(III) and Cr(VI) ($r > 0.9999$).

The ICPMS-2030 connected to our Prominence Inert HPLC System enabled precise detection of hexavalent chromium in food packaging.

QUANTITATION OF ARSENIC SPECIES IN WHITE RICE AND BROWN RICE

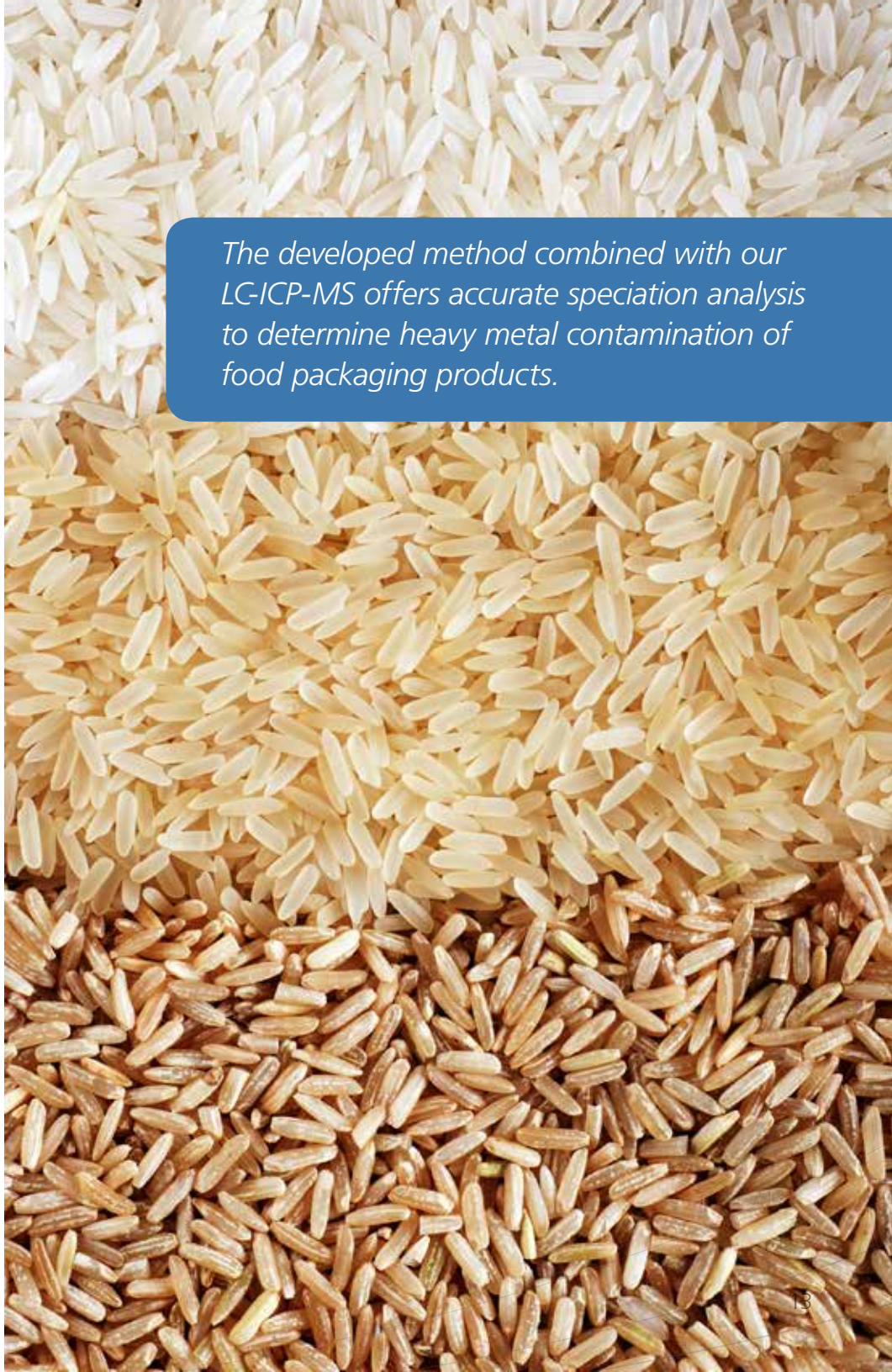
Rice contains a significant amount of arsenic with a high ratio of inorganic to organic arsenic. Both inorganic and organic arsenic species are known for their adverse health effects. However, inorganic arsenic is more toxic than organic arsenic.

One of the most efficient and reliable systems for detecting arsenic species is an LC-ICP-MS. Shimadzu's LC-ICP-MS system connects the ICPMS-2030 with a high-performance liquid chromatography (HPLC) system. Together, these two instruments offer higher sensitivity and improved accuracy.

Using our LC-ICP-MS system, we found arsenic concentrations in white and brown rice samples that were less than those certified by the National Metrology Institute of Japan. The results show good reproducibility with an RSD of 1.1% to 2.6%. The developed method combined with our LC-ICP-MS offers accurate speciation analysis to determine heavy metal contamination of food packaging products.

Content in Sample (mg/kg)		As (III)	As (V)	DMAA
White Rice	Quantitation Value (n=3)	0.0649	0.0203	0.0138
	Total of Inorganic Arsenic Species	0.0852		-
	NMIJ Certified Value	0.0841 As (III)=As (V)		0.0133
	Expanded Uncertainty	0.003		0.0009
Brown Rice	Quantitation Value (n=3)	0.254	0.0498	0.0186
	Total of Inorganic Arsenic Species	0.303		-
	NMIJ Certified Value	0.298		0.0186
	Expanded Uncertainty	0.008		0.0008
	RSD (%) (n=6)	1.1		2.6

Table 5: Measurement results for arsenic species

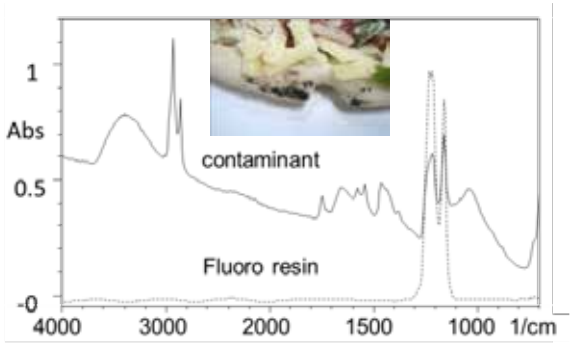


The developed method combined with our LC-ICP-MS offers accurate speciation analysis to determine heavy metal contamination of food packaging products.

EDX-FTIR CONTAMINANT FINDER/MATERIAL INSPECTOR (EDXIR-ANALYSIS) SOFTWARE

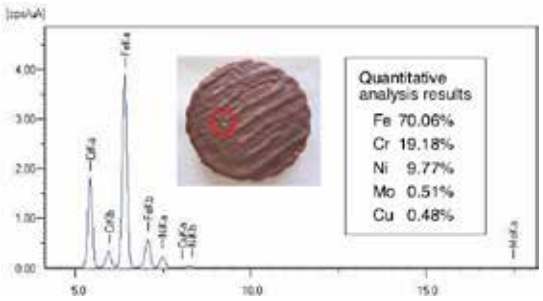
Energy dispersive X-ray (EDX) fluorescence spectrometry is used to perform qualitative and quantitative analysis of samples by measuring re-emitted characteristic X-rays from elements. Shimadzu's EDX-7000 and EDX-8000 can analyze samples in a variety of conditions, including solid, liquid and powder form, without requiring pretreatment.

For more efficient contaminant analyses and confirmation tests, Shimadzu offers the EDXIR-Analysis software package. This software allows you to integrate and analyze data acquired from an EDX fluorescence spectrometer and a Fourier transform infrared spectrophotometer (FTIR). EDX fluorescence spectroscopy is ideal for the elemental analysis of metals, inorganic compounds and other content, while an FTIR spectrophotometer is designed for the identification and qualification of organic compounds. The EDXIR-Analysis software compiles data from both instruments, allowing the use of our expansive library database to measure and qualify both inorganic and organic food contaminants.



Analysis of Foreign Object on the Frozen Pizza

With over 485 data acquired as standard, our library contains all the contaminants currently available. Additional data can be registered to the library along with image files and document files in PDF format. The library is also effective for the linked storage of various types of data as electronic files.



Metal Foreign Matter on Chocolate

With over 485 data acquired as standard, our library contains all the contaminants currently available.

Accurately detecting heavy metals is critical to ensuring compliance with the FDA's Food Safety Modernization Act (FSMA) and, more importantly, the safety of consumers. Even small traces of toxic metals can have serious health consequences. Shimadzu offers advanced instrumentation that provides the high-quality results you need to keep your lab running efficiently.

REFERENCES

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Frequently Asked Questions on FSMA:
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FSMA Final Rule for Preventive Controls for Human Food:
https://www.fda.gov/Food/GuidanceRegulation/FSMA/ucm334115.htm

To learn more about food contaminant testing using Shimadzu instruments, visit
www.FeedYourLab.com

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