SIMULTANEOUS DETERMINATION OF VARIOUS ELEMENTS IN INFANT FORMULA USING ICP-MS 2030

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1. Overview

Infant formula market represented more than 30 billion dollars in the world (2015)[3]. 46% of infant formula are produced in Europe and North America, Asia accounted for 49% of consumption. Like all the food products, strict regulation exists to ensure the infant formula quality. In Europe, national rules refer to European directives No 2006/141/CE and 2006/125/CE, which describe minimum and maximum levels for trace elements (Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, P, Se and Zn) in infant formula to ensure child growth up. At the same time, Commission regulation No 1881/2006 is setting maximum levels for contaminants (Al, As, Cs, Hg, Pb, Sb and Sn) in foodstuff and especially in baby food in order to prevent children from toxic substances.

Infant formula elemental analysis constitute a real challenge for laboratories because of the different concentration levels (from ppb to hundred ppm) of each targeted element.
Then a fast and easy to use method for simultaneous analysis of 19 elements (Al, As, Ca, Cr, Cs, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, P, Pb, Sb, Se, Sn and Zn) in infant formula has been developed using the Shimadzu ICPMS-2030.

2. Shimadzu ICPMS-2030: a valuable system for infant formula analysis

Thanks to the detector, ICPMS-2030 associates high sensitivity (trace detection) with a wide dynamic range (10^9) which is the key for simultaneous determination of major and trace elements.

Due to the unique Eco-Mode associated with Minitorch, ICPMS-2030 is able to drastically reduce running costs by half. The octopole collision cell assures a high accuracy for all elements measured. Using Helium gas and Kinetic Energy Discrimination principle (KED), this cell suppresses most of the spectroscopic interferences (polyatomic interferences). The efficiency of interferences suppression and enhancement of sensitivity are improved by a cooled cyclonic chamber and well controlled torch positioning.

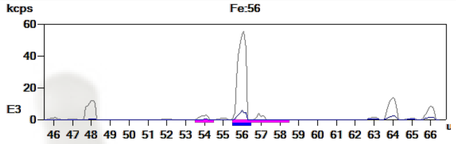


Figure 1: Development assistant window in LabSolutions ICPMS for ^{56}Fe in infant formula sample (INF1). Grey curve correspond to DBN mode and blue to DBG measurement mode.

Moreover, ICPMS-2030 is able to save all the masses from samples measurements and allowing results treatment without need of new analysis. Then thanks to its exclusive development assistant, *LabSolutionsICPMS* software is able to propose the optimum parameters for each element in the sample. Method development has never been so easy and fast than with ICPMS-2030.

Parameter	Setting
RF generator power	1.2 kW
Plasma gas	8 L/min
Auxilliary gas	1,1 L/min
Carrier gas	0.7 L/min
Nebulizer type	MicroMist
Sampling depth	5 mm
Spray Chamber temperature	3°C
Coll. Cell gas flow (He) DBG mode only	6 mL/min
Quantified Isotopes (contaminants/ trace elements)	^{27}Al, ^{75}As, ^{43}Ca, ^{53}Cr , ^{133}Cs, ^{63}Cu, ^{56}Fe, ^{198}Hg, ^{39}K, ^{25}Mg, ^{55}Mn, ^{95}Mo, ^{23}Na, ^{31}P, ^{206}Pb, ^{121}Sb, ^{78}Se, ^{118}Sn, ^{66}Zn
Internal Standards (ISTD)	^{113}In, ^{45}Sc, ^{159}Tb, ^{89}Y

Table 1: ICPMS-2030 measurement parameters.

Two different infant formula preparations, called INF1 and INF2, are analyzed. A microwave assisted acid dissolution is conducted, using CEM Mars™ 6.

0,5 g of each sample is treated by 3 mL of nitric acid and 2 mL of distillated water. Moreover 1 mL of Au 1 ppm solution is added to prevent Hg losses during mineralization process. Finally, after microwave treatment, the resulting clear solution is completed at 50 mL with distillated water before ICP analysis.

4. Calibration

For each studied element, calibration curves include 5 or 6 points in the targeted concentration range are realized in a 6% nitric acid solutions.

Infant formula samples are measured in triplicate and one of them, INF1 is spiked with known concentration values (depending target element level) in order to testify method accuracy.

An internal standard solution in 6% nitric acid was mixed using online mixing with sample, before it is aspirated (^{69}Ga, ^{71}Ga, ^{115}In, ^{205}Tl). Note that ISTD and their concentrations are automatically chosen by *LabSolutionICPMS* software for each studied element.

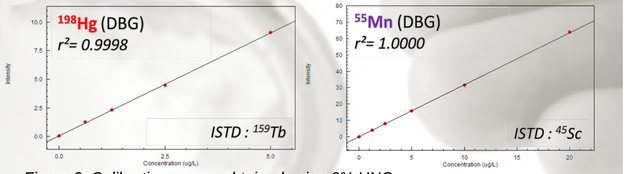


Figure 2: Calibration curves obtained using 6% HNO_3.

5. Detection limits calculation

Detection limits (LD) are calculated automatically by *LabSolutionICPMS* software with 3σ method.

Element	^{43}Ca	^{53}Cr	^{63}Cu	^{56}Fe	^{39}K	^{25}Mg
LD (ppb)	1.6	0.032	0.056	0.12	1.6	0.61
Element	^{55}Mn	^{95}Mo	^{23}Na	^{31}P	^{78}Se	^{66}Zn
LD (ppb)	0.001	0.008	1.5	1.2	0.048	0.01

Table 2: LD values for infant formulas trace elements.

Element	^{27}Al	^{75}As	^{133}Cs	^{198}Hg	^{206}Pb	^{121}Sb	^{118}Sn
LD (ppb)	0.14	0.002	8.10^{-4}	0.016	0.002	0.002	0.026

Table 3: LD values for infant formula contaminants

Low values of detection limits (LD) showed in Tables 2 and 3 indicate the high suitability and ability of ICPMS-2030 analysis infant formulas matrix.

6. Results

For the two infant formula studied results are synthesized in Table 4 and 5 for trace elements and contaminants respectively.

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 4: Trace elements concentration in studied infant formula (ppm; ** ppb).*

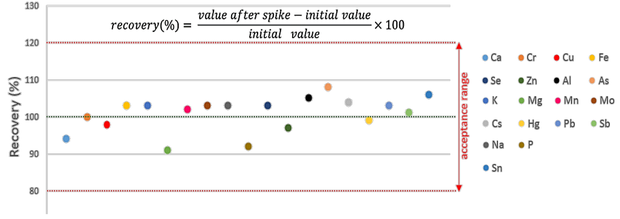
Element (ppb)	^{27}Al	^{75}As	^{133}Cs	^{198}Hg	^{206}Pb	^{121}Sb	^{118}Sn
INF1	894	4.1	9.7	2.7	5.9	3.0	18.9
INF2	1249	5.4	21.2	2.3	3.1	2.4	18.2

Table 5: Contaminants concentration in studied infant formula

The quantitation results in table 4 and 5 demonstrate that ICPMS-2030 is able to quantify simultaneously the various elements present in infant formula samples.

7. Method accuracy

Some spike of each element is done in infant formula INF1. The achieved results are displayed in graph 1.



Graph 1: recovery values for each element in INF1

The recovery rate for all the elements are between 91% and 107% indicating the suitability of the developed method in point of accuracy, independent from initial element concentration.