Application Brief

Food

Extending ICP-MS Capabilities to Detect Nanoparticles in Food

Monitor nanoparticles in infant formula using single particle (sp) mode on the Agilent 7800 ICP-MS

Authors

Shuofei Dong and Michiko Yamanaka

Agilent Technologies, Inc.

Measure multiple elements in nanoparticles in addition to your routine multi-element analysis

Due to its speed, multi-element capability, low detection limits, and wide dynamic range, the Agilent 7800 ICP-MS is widely used for routine analysis of food products. However, changes to food safety regulations and rising consumer awareness have increased demand for nanoparticle (NP) monitoring in food, in addition to the determination of the total elemental content.

Nanoparticle analysis of infant formula by spICP-MS

In this study, the 7800 ICP-MS was fitted with the standard MicroMist glass concentric nebulizer, quartz Scott-type spray chamber, and nickel interface cones. The standard torch was replaced with a quartz torch with a 1.0 mm i.d. injector, as usual for NP applications. The optional Single Nanoparticle analysis and Advanced Acquisition modules were added to the ICP-MS MassHunter software to enable fast time resolved analysis (TRA) and integrated, multi-element single nanoparticle (sNP) data processing. Fast TRA mode uses 100 µs integration times with no settling time between measurements. This provides the optimum balance between sensitivity and acquisition speed.

Thirteen elements were selected for sNP monitoring, based on the potential major and trace element content of NPs in infant formula. The ORS^4 collision/reaction cell was operated in no gas mode for Ag, Ba, and Pb, and with helium (He) collision gas for Al, Si, Ca, Ti, Cr, Mn, Fe, Ni, Cu, and Zn. Gas modes and analyte masses are acquired sequentially within a single visit to the sample vial, providing high throughput multi-element NP screening without compromising data quality. After setting the optimum nebulizer gas flow for the 1.0 mm i.d. torch, the plasma and tune settings were optimized automatically using batch autotune.

Table 1. Typical 7800 ICP-MS operating parameters used for spICP-MS method.

	Helium Mode	No Gas Mode
RF Power (W)	1550	
Sampling Depth (mm)	8.0	
Nebulizer Gas Flow (L/min)	0.75	
Energy Discrimination (V)	5.0	
Helium Flow (mL/min)	5.0	0

Standards and samples: Agilent multi-element calibration standards were used to determine the response factor (cps/ppb) of each element. Seven infant formula products were bought from supermarkets in the USA (3 samples) and China (CHN, 4 samples). About 0.25 g of each sample was suspended in 25 mL de-ionized water (DIW) and then diluted a further 1:5 before analysis. A 60 nm Ag NP suspension (BBI Solutions, UK) was purchased to allow calculation of nebulization efficiency and to assess sample matrix effects.

Evaluation of matrix effects: To investigate the effect of the sample matrix on the NP signals, the same concentration of 60 nm Ag NP standard was spiked into DIW and the samples. The Ag NP particle concentration and size distribution were measured and compared. Figure 1 shows that there is no difference in the two Ag NP size distribution in the infant formula samples. Also, the Ag NP concentration recovery was > 87% compared with the spiked DIW, confirming that the sample matrix did not cause any significant matrix effects.

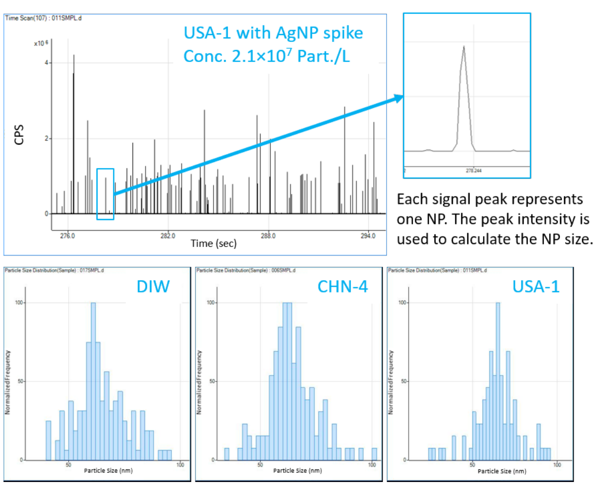

Figure 1. Ag NPs in spiked infant formula (USA-1, top left); detail showing signal for one NP (top right); comparison showing consistent Ag NP particle size distribution in DIW and two infant formula samples (bottom).

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Identifying multi-element NP content

NPs in food could arise from raw materials, manufacturing equipment, and/or packaging materials. The spICP-MS method was used to assess the presence of NPs containing the 13 elements of interest. NPs containing Al, Ca, Fe, and Zn were identified in some of the infant formula samples. Differences in the NP particle type and number were observed between the different infant formula samples, as illustrated in Figure 2 for Ca and Fe in two of the samples.

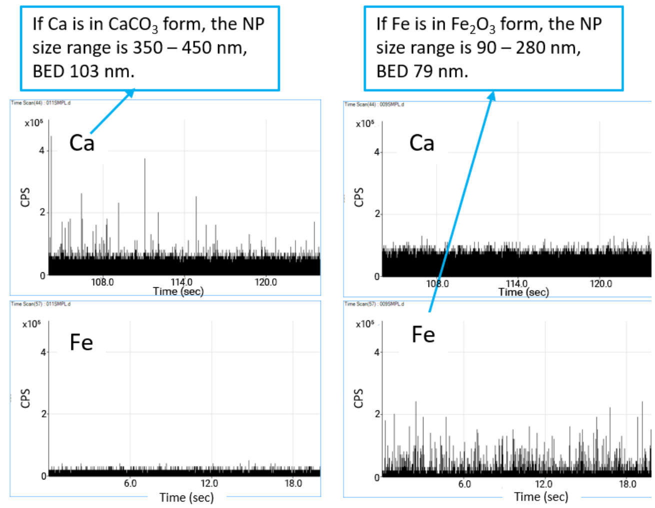

Figure 2. Examples of fast TRA data from multi-element NP analysis of infant formula samples. Comparison of size and number of Ca- and Fe-containing particles in USA-1 (left) and CHN-4 (right).

In addition to identifying the type and number of NPs, the size distribution can be estimated, even if the actual form of the element in the NPs is not known. NP size (diameter) can be calculated by assuming the most likely form of the element in the different NPs, such as $CaCO_3$ for Ca and Fe_2O_3 for Fe. Using these assumptions, the Ca NPs in infant formula USA-1 were calculated to be between 350 and 450 nm diameter, with a background equivalent diameter (BED) of 103 nm. The Fe NPs in infant formula CHN-4 were calculated to be between 90 and 280 nm diameter, with a BED of 79 nm.

Extending the capabilities of ICP-MS

The 7800 ICP-MS can be used routinely for high-throughput, accurate, quantitative measurement of major and trace elements in food samples. With the addition of optional software modules to support spICP-MS mode, the same instrument can be used to assess the multi-element NP content of samples such as infant formula. This flexibility enhances the usability of the 7800 ICP-MS to meet the increasing demand from the food industry for NP analysis in quality control and authenticity studies.

Printed in the USA, February 24, 2020
5994-1748EN
DE.8862037037

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