Analysis of milk powders based on Chinese standard method using the Agilent 5100 SVDV ICP-OES

Application note

Food Testing

Authors

Neli Drvodelic

Agilent Technologies
Melbourne, Australia

Introduction

The elemental composition of milk is a good indicator of environmental contamination. It is also a significant pathway for toxic metal intake as well as a source of essential nutrients for humans [1]. As such, standard methods and guidelines are being developed to monitor the elemental content of milk and milk-based products. For example, China's National Food Safety Standard, GB 5413.21—2010, covers the determination of calcium, iron, zinc, sodium, potassium, magnesium, copper and manganese in foods for infants and young children, raw milk and dairy products.

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Many of the major nutrients in milk powder such as Na, K and Ca are present at elevated levels. Some toxic elements must be determined at low levels, which means instrument sensitivity is of paramount importance. This drives the analysis towards an axial ICP-OES instrument in order to reach the low quantitation limits required for the toxic elements, but strategies to measure the higher levels of major nutrient elements must also be considered. Previous work [2, 3] has shown that the addition of caesium (Cs) as an internal standard and ionization suppressant to the standards and samples was beneficial in measuring the higher levels of elements such as Na and K. An alternative strategy to cover this large measurement range, is to measure the trace elements axially, and the nutrient elements radially using a Dual View (DV) ICP-OES instrument. However, conventional DV instruments take two or more sequential measurements of the sample for a complete analysis of all elements.

In this work, the Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES with unique Dichroic Spectral Combiner (DSC) technology was used to measure both axial and radial light in one reading, enabling major elements such as Na and K to be measured radially while trace elements are measured axially [4]. This extends the upper measurement range, and reduces interferences for nutrient elements like Na and K, and at the same time, enables trace elements like Zn, Cu and Mn to be determined, with no time penalty, low argon consumption per sample, accurate and precise data and an exceptional linear dynamic range (LDR).

The experimental work was carried out in accordance with China's GB 5413.21—2010 standard method and the accuracy and validity of the method was assessed by analyzing National Institute of Standards and Technology (NIST) 8435 Whole Milk Powder Standard Reference Material (SRM).

Instrumentation

All measurements were performed using an Agilent 5100 SVDV ICP-OES with DSC that runs axial and radial view analysis at the same time. If greater flexibility of operation is required, the 5100 SVDV ICP-OES can be run in four different modes: synchronous vertical dual view, vertical dual view, radial and axial. All 5100 configurations feature a vertical torch and 27 MHz free running solid state RF generator, allowing the user to measure the most challenging samples, including high matrix food samples, with less cleaning, less downtime and fewer replacement torches.

The sample introduction system consisted of a Seaspray nebulizer, double-pass glass cyclonic spray chamber and a standard 1.8 mm torch. An SPS 3 autosampler was used to deliver samples to the instrument. Yttrium was used as an internal standard and added online via a tee piece.

The instrument operating conditions used are listed in Table 1.

Table 1. The Agilent 5100 SVDV ICP-OES operating parameters used.

Parameters	Settings
Read time (s)	10
Replicates	3
Sample uptake delay (s)	20
Stabilization time (s)	15
Rinse time (s)	30
Pump Speed (rpm)	15
Fast pump (rpm)	80
RF power (kW)	1.3
Aux flow (L/min)	1.0
Plasma flow (L/min)	12
Nebulizer flow (L/min)	0.7
Viewing height (mm)	6
Sample pump tubing	White-white
Internal standard tubing	Orange-green
Internal Standard	Y 371.029 for all elements

Experimental

Standard and sample preparation

NIST SRM 8435 Whole Milk Powder was used as the sample during this study. Concentrated HNO_3 (7 mL) and 30% H_2O_2 (1 mL) were added to approximately 1.0 g of the milk powder SRM and digested in a laboratory microwave. Although the GB 5413.21—2010 method outlines an ashing/digestion sample preparation procedure, microwave digestion is also acceptable. Once dissolved, the solutions were allowed to cool and made up to volume in a 25 mL volumetric flask. The final acid concentration was approximately 20% v/v HNO_3 and the final dilution was 1:25.

Multi-element calibration standards were prepared from single stock solutions per the GB method. Standard working solutions of different concentrations for the different elements were prepared per the concentrations listed in Table 2. The calibration range for Na, K, Mg, and Ca was extended , which highlights the large LDR of the 5100 with SVDV. The GB method permits the manual dilution of samples outside the calibration range, but this wasn't required with the excellent linear dynamic range of the 5100 SVDV ICP-OES.

Results and discussion

The Method Detection Limits (MDL) were based on three sigma of ten replicate measurements of the blank solution during the analytical run. As can be seen in Table 3, the MDLs obtained on the 5100 SVDV ICP-OES are below those specified in the GB method.

Table 3. Method detection limits acquired per GB 5413.21—2010 guidelines. All MDLs were determined in a single analytical run.

Element	MDL (mg/kg)	MDL (mg/100g)	GB Specified DL (mg/100g)
K 766.491	1.74	0.17	0.7
Ca 315.887	0.10	0.01	0.7
P 213.618	0.17	0.02	N/A
Na 589.592	0.08	0.01	1.6
S 181.972	1.11	0.11	N/A
Mg 279.078	0.08	0.01	0.2
Zn 202.548	0.006	0.0006	0.002
Sr 421.552	0.0005	0.00005	N/A
Fe 259.940	0.01	0.001	0.003
Cu 327.395	0.01	0.001	0.002
Mo 204.598	0.03	0.003	NA
Mn 257.610	0.003	0.0003	0.005

Table 2. Composition and concentration of mixed standard concentration solution, µg/mL

Calibration Standards	Fe	Mn	Cu	Mo	Sr	Mg	Zn	P	S	Ca	K	Na
	Concentration (µg/mL)											
1	0.05	0.05	0.05	0.05	0.05	1	1	1	1	10	10	1
2	0.5	0.5	0.5	0.5	0.5	5	5	10	10	50	50	10
3	1	1	1	1	1	10	10	100	100	100	100	20
4	5	5	5	5	5	25	25	200	200	250	250	50
5						50	50	500	500	500	500	100
6										750	750	150
7												200

The recoveries for the elements determined in the milk powder SRM were in the range of 92% to 107% highlighting the accuracy of the method. The results in Table 4 also demonstrate the wide dynamic range capability of the 5100 SVDV ICP-OES, as elements were determined at the ppb to % level in a single reading.

Although the Chinese GB method currently covers the determination of eight elements (Ca, Fe, Zn, Na, K, Mg, Cu and Mn), results for P, S, Sr and Mo in the SRM have been reported as they may be important for milk powder analysis in other countries or regions.

Table 4. Analysis of NIST Milk Powder 8435 SRM using the 5100 SVDV ICP-OES.

Element	Certified value (mg/kg)	Measured values (mg/kg)	Recovery (%)
Major nutrients			
K 766.491	13630	13070	96
Ca 315.887	9220	9750	106
P 213.618	7800	7160	92
Na 589.592	3560	3530	99
S 181.792	2650	2650	100
Minor and trace nutrients			
Mg 279.078	814	749	92
Zn 202.548	28.0	28.9	103
Sr 421.552	4.35	4.37	101
Fe 259.940	1.8	1.9	107
Cu 327.395	0.46	0.46	100
Mo 204.598	0.29	0.27	92
Mn 257.610	0.17	0.18	103

Conclusions

The Agilent 5100 SVDV ICP-OES with DSC technology allows measurement of both axial and radial readings in a single, fast, cost effective measurement. Trace toxic and major nutrient elements were measured in a single measurement in a milk powder SRM, with no ionisation buffers. Excellent recoveries were achieved for all elements determined in the SRM using SVDV mode demonstrating the accuracy of the method over a large dynamic range. This work shows that the Agilent 5100 SVDV ICP-OES is suited to Chinese method GB 5413.21 for the analysis of milk powders.

References

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