Application Brief

Food Testing and Agriculture

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Rapid Determination of Seven Elements in Milk Powder by Microwave Digestion and Flame Atomic Absorption Spectrometry

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Introduction

Flame Atomic Absorption Spectrometry (FAAS) can be used to determine the concentration of nutrient elements in milk and milk products. It is a low cost and simple technique to own and operate. Being a long established analytical technique, FAAS is included in many standard methods and regulations, so the analytical parameters and requirements are well documented.

FAAS analysis can be slow, compared to newer techniques such as ICP-OES. Samples must be measured multiple times—once for each element being measured. This makes the technique inefficient and may introduce errors.

There have been techology developments that have improved the efficiency of FAAS. In this study, seven elements; K, Na, Ca, Mg, Fe, Zn and Cu, were determined in milk powder from a single sample measurement with an Agilent 280FS Flame AAS using Fast Sequential (FS) mode.

Fast Sequential mode

The Agilent 280FS is a high performance flame atomic absorption spectrometer. It combines eight lamps with the Agilent Fast Sequential mode which doubles sample throughput and dramatically reduces running costs. The 280FS is ideal for labs wanting the best performance.

Fast Sequential mode operates all eight lamps simultaneously. A fast lamp selection mirror and high speed wavelength drive are used to switch between the lamp and wavelength required to measure each element. The mode allow the rapid determination of the seven nutrients in milk powders from a single sample aspiration into the flame. This compares to conventional FAAS where only one element is measured with each aspiration of a sample—measuring seven elements would require the sample to be presented to the instrument seven times, increasing analysis time, gas consumption and the risk of errors. FS mode saves time, reduces gas consumption and occurrence of operator error.

Instrument setup

Seven single element, Agilent-coded, hollow cathode lamps were used for this analysis: K, Na, Ca, Mg, Fe, Zn, and Cu.

All the glassware for the experiment were soaked in nitric acid (1 in 4 dilution) for 24 h, rinsed with distilled water and then with deionized water, and air dried for later use.

Calibration standards are shown in Table 1.

Table 1. Concentrations of standards (mg/L).

Element	1	2	3	4	5
K	0.050	0.10	0.20	0.40	0.80
Na	0.050	0.10	0.20	0.40	0.80
Ca	0.50	1.0	2.0	4.0	8.0
Mg	0.050	0.10	0.20	0.40	0.80
Cu	0.50	1.0	2.0	4.0	8.0
Fe	0.50	1.0	2.0	4.0	8.0
Zn	0.10	0.20	0.50	1.0	2.0

The instrument settings are shown in Table 2. The blank and standard solutions were measured to create calibration curves. The correlation coefficient of the standard curve for each element is shown in Table 3.

Table 2. Instrument settings.

Element	Wavelength (nm)	Slit Width (nm)	Lamp Current (mA)	Air Flow Rate (L/min)	Acetylene Flow Rate (L/min)
K	766.5	0.5	5	13.50	2.0
Na	589.0	0.5	5	13.50	2.0
Ca	422.7	1.0	10	13.50	2.0
Mg	285.2	0.2	4	13.50	2.0
Cu	324.8	0.5	10	13.50	2.0
Fe	248.3	0.2	10	13.50	2.0
Zn	213.9	0.2	4	13.50	2.0

Table 3. Correlation coefficient of the standard calibration curve created for each element.

Element	Correlation coefficient
K	0.9991
Na	0.9987
Ca	0.9994
Mg	0.9996
Cu	0.9998
Fe	0.9989
Zn	0.9993

Microwave digestion procedure

0.500 g of the sample was weighed into a microwave digestion vessel into which 5 mL of nitric acid was then added. The sample was then placed in a microwave digestion system, using the following conditions: Heating to 120 °C in 5 min and then holding for 5 min; heating to 160 °C in 5 min and then holding for 10 min; heating to 180 °C in 5 min and then holding for 10 min. The acid was then boiled off at 140–160 °C until the sample volume was reduced to 1 mL. The sample was transferred into a 50 mL volumetric flask, with the digestion vessel being rinsed 2 to 3 times with a small amount of water. The rinse solutions were added to the volumetric flask and 5.0 mL of lanthanum oxide solution was added. The final dilution factor was 100.

Precision and accuracy

Six replicates of 0.500 g of a milk powder standard reference material were accurately weighed, digested using a microwave digestion system, and analyzed. Results are shown in Table 4.

Excellent recoveries (within ±10%) were obtained for each of the seven elements, based on the certified values of the reference material. The RSD% was <2.5% for each element, except Ca, which was 3.7%.

The results demonstrate the high accuracy and precision of the 280FS when using FS mode.

Table 4. Precision and recovery (n = 6).

Element	Measured value (mg/ 100 g)	RSD (%)	Certified value (mg/ 100 g)	Mean recovery (%)
K	693.6	2.35	717.0	97
Na	141.5	1.48	157.0	90
Ca	628.2	3.67	616.5	102
Mg	67.41	0.74	70.94	95
Cu	418.9	0.32	434.5	96
Fe	7.214	1.01	7.440	97
Zn	4.027	0.63	4.310	93

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