application note

Nutritional and Micro Nutritional Analysis of Milk Powders and other Dairy Products using the Slurry Method of Sample Introduction

Introduction

Nutrition in the early stages of life depends entirely on milk, be it human or a formula product. Milk and milk products also provide a major part of adult nutrition. As milk is also added to a wide range of food products, it is not surprising that this food source is being increasingly investigated by national health authorities and international organisations.[1–3] Both investigative and regulatory steps have been undertaken to ascertain actual dietary intakes of nutritionally important elements such as zinc, magnesium, phosphorus and calcium, as well as major and other trace elements in milk.

The traditional method of determining the nutritional composition of milk is to ash it at high temperatures, remove the organic matter and then follow this with acid digestion. The digestate is then quantitatively transferred into a volumetric flask and made up to volume with distilled/deionised water. This ashing and digestion procedure can take up to a day to perform (the ashing is usually done overnight). As well as the time costs involved, this procedure can lead to some loss of volatile elements during the ashing stage and it also increases the possibility of introducing contaminants in the digestion stage.

This report presents an alternative simplified approach to the determination of milk and related products. The benefit of using the milk slurry technique in preference to the traditional ashing or digestion methods is also discussed.

Experimental

A GBC Integra ICP-OES was used for the analysis of the milk digests and milk slurries. The spectrometer fitted with the optional 1800 groove/mm holographic grating was employed. This provided typical resolution of 0.018 nm in 1st order, 0.009 nm in 2nd order, 0.007 nm in 3rd order and 0.006 nm in 4th order.

The Integra has Mass Flow Sensors fitted to the sample, plasma and auxiliary gas lines. Having the Mass Flow Sensor fitted to the sample gas is advantageous in the analysis of samples with high dissolved salt contents. It provides direct continuous monitoring of the nebulizer pressure, essential for the immediate diagnosis of nebulizer blockages. This parameter was monitored continuously throughout the duration of the analysis conducted for this report.

The set of sample introduction components used were the V-groove nebulizer, low volume rapid wash cyclonic spraychamber and unique three piece demountable torch.

This report focuses on the comparison of the analysis of the milk ash/digest with the milk slurry.

The operating conditions used for the analysis of the milk slurries are presented in Table 1.

The power, viewing height and sample gas flows were optimized to obtain the best Signal to Background Ratio (SBR) using the unique Auto Optimization Simplex Algorithm.

Table 1: Instrument Operating parameters used

Power (w)	1200
Plasma Gas (L/min)	11.00
Auxiliary Gas (L/min)	0.50
Sample gas (L/min) Sample gas (kPa)	0.50 280
Torch height (mm)	9.0
Pump speed (mL/min) Pump speed (rpm)	1.2 15
PMT (v)	500
Scan Window	0.08 nm 1st order 0.04 nm 2nd order 0.027 nm 3rd order 0.02 nm 4th order
Data Points	40
Integration Time Per Point(s)	0.1
On Peak Integration Time (s)	2
Replicates	3
Background Correction Mode	Dynamic
Fast pump between samples (s)	15
Stabilization time (s)	20

Sample and Standard Preparation

Slurry Sample Preparation

Nestlè Australia provided a milk powder which they globally use as a reference check sample.

3.00 g of sample was accurately weighed into a 100 mL volumetric flask and made up to volume with distilled/deionised water. The slurry samples were shaken for 20 seconds prior to aspiration.

Sample Ash/Digestion

A 10 mL aliquot was dried after adding 1 mL HNO_3 (30%) heated and ashed. The ashing took place at 450°C overnight. To the white ash 2 mL HNO_3 (30%) was added and made up to 100 mL with water and analysed.

Standard Preparation

All standards were prepared from high purity 1000 ppm stock standard solutions acidified by the supplier with 0.5 M HNO_3.

Two multi element standards and a blank were prepared in order to ensure linearity over the expected concentration range for each element.
The selected wavelengths and concentrations of the standards are presented in Table 2.

Table 2: Analytical wavelengths and calibration standards used

Element	Wavelength (nm)	Standard 1 (ppm)	Standard 2 (ppm)
Na	589.592	10.00	50.00
K	769.896	25.00	100.0
Ca	422.673	25.00	100.0
Mg	280.270	2.00	10.00
Fe	238.204	0.20	2.00
Zn	213.856	0.20	1.00
Cu	324.754	100.0	500.0
Mn	259.373	20.00	50.00
P	177.495	15.00	30.00

Results and Discussion

Long Term Stability

The long term stability of the ICP was measured by continuously aspirating the milk slurry for over an hour. The results are plotted in Figures 1 and 2. The reproducibility of the measurements over 1 hour ranged from 0.74% RSD for Cu to 1.58% RSD for Ca.

Slurry versus Ash/Digest Results

The ashed sample and the 3% slurry were analysed in duplicate and the results are presented in Table 3. The values presented are calculated back to the solid.

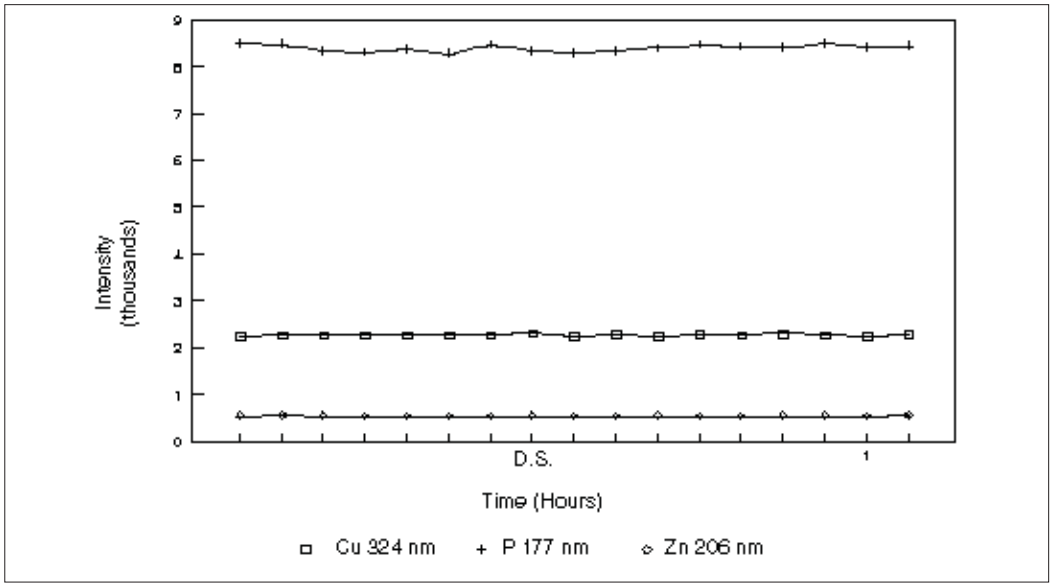

Figure 1 *Long term stability for milk slurry for Cu, P and Zn*

Table 3: Comparison of duplicate sets of results in ashed and 3% slurry samples for the reference milk powder

Element	Nestlè Reference Powder Expected Result (mg/100g)	Ashed Samples (mg/100g)	3% Slurry Results (mg/100g)
Na	116.9–126.4	115, 115.8	120, 120
K	588–710	680, 670	672, 675
Ca	273.3–284.8	278.3, 276.7	279.3, 278.9
Fe	24.7–26.7	25.1, 25.2	25.1, 25.3
Mg	3.9–4.15	3.92, 3.95	3.93, 3.95
Zn	1.50–1.65	1.52, 1.52	1.62, 1.62
Cu	0.125–0.150	0.128, 0.127	0.142, 0.142
Mn	30.6–50.1	40.9, 41.8	40.8, 40.9
P	215.8–224.3	226.8, 227.3	223.0, 222.8

Both the slurry and the ash digest samples fall within the Nestlè Reference Powder expected range. There is also good agreement between the slurry values and the ashed/digest values. Further, there is also very good agreement within the duplicate set of samples.

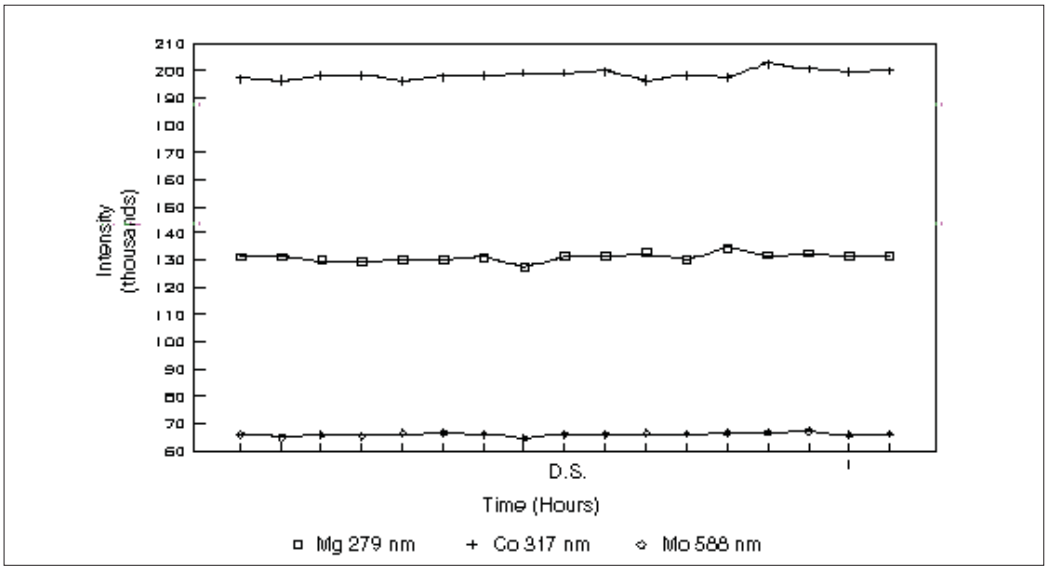

Figure 2 *Long term stability for milk slurry for Mg, Ca, and Na*

Conclusion

The Integra ICP offers an alternative simplified approach to the determination of milk and related products. The slurry method of sample introduction provides a very productive alternative to the traditional time consuming digestion method without compromising accuracy or reproducibility.

In summary, the benefits that the milk slurry technique has compared to the traditional ashing or digestion method are as follows:

- improved productivity with less time spent preparing a slurry compared to performing time consuming chemical digestion or ashing procedures,
- reduced likelihood of introducing contaminants from acids and other reagents,
- more accurate results with volatile elements which may be lost during a digestion or ashing procedure, and
- significant cost savings in terms of equipment, reagents and operator time.

With the elimination of time consuming digestion methods, the GBC Integra ICP-OES increases productivity without compromising performance.

Acknowledgment
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References

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GBC Scientific Equipment Pty Ltd
A.C.N. 005 472 686

12 Monterey Road, Dandenong, Victoria, 3175, Australia
Phone: +61 3 9213 3666 Fax: +61 3 9213 3677
E-mail: gbc@gbcsci.com URL: www.gbcsci.com