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The determination of major and minor elements in milk and infant formula by slurry nebulisation and inductively coupled plasma - optical emission spectrometry (ICP-OES)

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Abstract

A method for the analysis of Ca, Cu, Fe, K, Mg, Mn, Na, P and Zn in infant formulas, milkpowders and liquid milk was developed using ICP-OES. Samples were prepared as slurries and determined against aqueous standards following Lu internal standard compensation for potential matrix effects. The instrument used was radially configured and incorporated a charge-transfer device detector allowing simultaneous acquisition of multi-element and multi-line measurements. Instrument and method performance parameters were estimated for precision, linearity, background equivalent concentrations, detection limits and accuracy. The method was found to be suitable for routine quality control monitoring of infant formulas and milkpowders, thereby improving sample throughput and analytical confidence.

1. Introduction

It is acknowledged that the composition of infant formulas requires rigorous regulatory control (Tanner & Barnett, 1985; Ruiz et al., 1995), with specific concerns related to mineral content (Gillies & Neill, 1985). Thus, in the determination of elemental composition of infant formulas, the emphasis has justifiably focussed on accuracy and precision, with sample throughput of secondary concern. However, the conundrum of any quality control (QC) laboratory is to analyse compliance samples with minimal cost, while retaining an appropriately high degree of confidence in results generated.

Many techniques have been utilised for the elemental analysis of a range of matrices, including colourimetry, polarimetry, stripping voltammetry, X-ray fluorescence, neutron activation analysis, capillary zone electrophoresis, and complexometry (Tolg, 1987; Skurikhin, 1989; Lavi & Alfassi, 1990; Bersier et al., 1994; Pretswell et al., 1995). Several of these strategies have been utilised primarily for trace element speciation (Florence, 1986), as well as in the collaborative certification of international reference milk powders (Griepink et al., 1984). However, atomic spectrometry has become established as the mainstay of trace element analysis in foods, with flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS), and flame emission spectrometry (FES) the predominant techniques in use (Brown et al., 1989). Recently however, the maturation of inductively coupled plasma-optical emission spectrometry (ICP-OES) incorporating charge-transfer device (CTD) detectors has provided the opportunity to develop a flexible, user-programmable, simultaneous multielemental capability, facilitating increased spectral information and sample throughput. The low detection limits, high quantum efficiency, wide linear dynamic range (LDR) and inherent simultaneity of the CTD compared to photomultiplier detectors make ICP-OES a viable alternative to the aforementioned techniques (Hanley et al., 1996).

Sample preparation now remains the major limiting step in analytical throughput, a restriction emphasised by the multielement capabilities of modern ICP-OES instrumentation. Traditional methods of sample preparation for infant formulas, milkpowders and liquid milks are well documented. These include wet and dry ashing (Miller-Ihli, 1996; Arnaud et al., 1992; AOAC, 1995a; Vuchkova et al., 1996), microwave digestion (de la Fuente et al., 1995; Borkowska-Burnecka et al., 1996), and lyophillisation followed by ashing (Coni et al.,

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1994, 1996). An alternative technique of which relatively little has been reported is the slurry nebulisation technique, involving the direct aspiration of diluted milk, reconstituted powdered milk powder or infant formula directly into an AAS, AES, or ICP-OES system. This has been reported as successful for the major mineral components of infant formulas (Ca, Mg, Na, K, Fe) by FAAS and FES (Ruiz et al., 1995; de la Guardia et al., 1986). An attempt to determine a range of trace minerals in human milk by slurry nebulisation into an ICP-OES system reportedly failed (Coni et al., 1990), although a recent paper (Nobrega et al., 1997) reports successful application of this combined methodology for Ca, Mg, K, Na and P. A recent report has described the determination of Cu and I in reconstituted milk-powder by flow-injection ICP-mass spectrometry (MS) (Sturup & Buchert, 1996), while an interesting study of trace element speciation in milk has utilised direct injection of defatted milk onto a coupled HPLC–ICP-OES system (Bratter et al., 1998). Compared to conventional mineralisation based procedures, a slurry nebulisation protocol offers inherent benefits of simplicity and reduction of sample manipulation, thereby minimising the potential for losses or contamination. An excellent review of the technique in conjunction with ICP has recently been published (Ebdon et al., 1997).

This paper reports the development of a facile and convenient sample preparation scheme for the determination of Ca, Mg, Na, K, Fe, Zn, Cu, Mn and P in infant formulas and milks following slurry nebulisation of diluted reconstituted powder or fluid milk into a radially configured ICP-OES, with quantification incorporating lutetium internal standardisation.

2. Materials and methods

2.1. Equipment

The ICP-OES instrument employed was a radial configuration OPTIMA 3000 (Perkin–Elmer, Norwalk, CT USA), incorporating a solid state segmented-array charge-coupled device (SCD) detector, Scott type spray chamber, and cone-spray high solids nebuliser, controlled with PE Winlab software, and configured with an AS90 autosampler (Perkin–Elmer). Other equipment used included calibrated 25 ml stoppered ground glass graduated test tubes, 100–1000 μl autopipette (BRAND, Germany), and volumetric glassware.

2.2. Reagents

All water was E-Pure ($\geqslant$18 MΩ). Individual stock standards of Na, K, Ca, Mg, P (10 000 ppm), and Fe, Zn, Cu, Mn, Lu (1000 ppm) were ICP grade (AristaR, BDH, Poole, UK). Concentrated nitric acid (AnalaR, BDH) was diluted to 0.5% v/v with water, and Triton X-100 (BDH) diluted to 10% v/v with water.

2.3. Standard preparation

Two intermediate multielement standards were prepared containing (i) Ca (400 ppm), K (400 ppm), Na (100 ppm), Mg (40 ppm) and (ii) Fe (40 ppm), Zn (20 ppm), Cu (2.0 ppm), Mn (1.0 ppm). An intermediate Lu standard (50 ppm) was prepared separately. Two multielement working standards were then prepared: (i) Ca (200 ppm), K (200 ppm), Na (50 ppm), Mg (20 ppm), Lu (2.0 ppm) and (ii) Fe (2.0 ppm), Zn (1.0 ppm), Cu (100 ppb), Mn (50 ppb), Lu (2.0 ppm). A single element P working standard (100 ppm) containing Lu (2.0 ppm) was prepared, and stabilised with 0.5% HNO_3 (2 ml/100 ml). A standard blank containing Lu (2.0 ppm) was also prepared.

2.4. Sample preparation

Approximately 0.25 g of powdered milk or infant formula or 2.5 g of liquid milk or infant formula was accurately weighed into a 25 ml graduated test tube, and approximately 15 ml of warm (ca. 37°C) water was added. Tubes were capped and vortexed to dissolve sample. After standing for 10 min and cooling to room temperature, 1.0 ml of Lu standard (50 ppm) and 250 μl of Triton X-100 solution (10%) was added to each tube, and made to volume with water and mixed.

2.5. Analysis

Preliminary studies involved the evaluation of instrumental performance of the ICP-OES relative to instrument specifications. Performance criteria for the elements of interest included detection limit (DL), linearity, precision and background equivalent concentration (BEC) and were estimated for multiple lines under preliminary instrumental operating parameters, as listed in Table 1(a).

A multi-line spectral interference study was conducted with aqueous standards (100 ppm), prior to assessment of the overall slurry nebulisation method operating under routine instrumental conditions as described in Table 1(b) and applied to the major lines for all nine elements. For routine QC use, single level calibrations were established for each element based on multielement standards, followed by automated analysis of sample extracts. Standards and sample emission counts were acquired in triplicate, with software reduction to report means and standard deviations. The method precision, accuracy, recovery, and detection limits were determined.

A Triton X-100 solution (1% v/v) was aspirated at an elevated flow rate (4 ml/min) for 20 s between samples

Table 1
Instrument operating parameters for ICP-OES during (a) instrument assessment [a] and (b) routine use [b]

Parameter	(a)	(b)
RF power (W)	1100	1350
Nebuliser flow (l/min)	0.95	0.80
Auxiliary flow (l/min)	15	15
Plasma flow (l/min)	0.50	0.50
Sample flow (l/min)	1.5	1.8
Source equilibration time (s)	15	15
Viewing height (mm)	15	15
Background correction	Manual point selection	Manual point selection
Measurement processing mode	area	area
Auto integration (s, min–max)	10–20	5–10
Read delay (s)	40	40
Rinse delay (s)	10	20
Number of replicates	10	3

[a] Lines used were: Ca 315.887, *Ca 422.673*; *Cu 324.754*, Cu 224.700; Fe 239.562, *Fe 259.940*, Fe 238.204; *K 766.491*; Mg 285.213, *Mg 279.553*, Mg 280.270; *Mn 257.610*, Mn 260.569; *Na 588.995*, Na 589.592; P 178.221, P 177.428, *P 213.618*; Zn 202.548, Zn 206.200, *Zn 213.856*. Data acquired for all lines, although only principal line data reported for IDL, BEC and instrumental precision. Principal line indicated in italics.

[b] MDL, method precision, recovery and comparative data acquired and reported for principal lines as listed above.

and for 15 min at the conclusion of each sample set prior to shut-down of the instrument.

2.6. Comparative methodology

Comparative data for an in-house control sample was obtained with use of an SP9 FAAS/FES instrument (Pye-Unicam, Cambridge, UK) after a dry ashing-acid digestion sample preparation for Ca, Cu, Fe, K, Mg, Na, and Zn (AOAC, 1995b), while a 5100 GFAAS (Perkin–Elmer) and slurry sample preparation incorporating standard addition quantification was utilised for determining Mn (Wagley et al., 1989). A colourimetric method incorporating the molybdovanadate reagent and a UV-160 UV-Vis spectrometer (Shimadzu, Kyoto, Japan) was employed for P (AOAC, 1995c).

A range of infant formulas and milk samples was also subjected to ICP-OES analysis following mineralisation by dry-ashing (AOAC, 1995b) for comparison against the slurry protocol. These same infant formula samples were further analysed by both FAAS and ICP-OES subsequent to conventional acid digestion at an independent regulatory laboratory.

3. Results and discussion

There were no significant instrumental spectral interferences for any single element emission line when evaluated against the multielement standards at the 100 ppm level. Instrument detection limit (IDL) and method detection limit (MDL) data for each element were estimated and summarised in Table 2.

The IDL's (measured as aqueous standards) for each element were comparable to those specified by the instrument manufacturer under the recommended conditions. MDL's for Cu, Fe, Zn and Mn, measured in a low analyte level infant formula powder, were 1–3 orders below analyte levels expected in QC samples, while it was unnecessary to determine MDL's for Ca, Mg, Na, K, and P since infant formulas and milkpowders contain these elements in abundance. Although axially configured ICP is generally acknowledged to yield an enhancement of DL, the radial configuration has proven in this work to provide an acceptable compromise between sensitivity and robustness.

Background equivalent concentration is defined as the concentration of a solution that results in an analyte emission signal equivalent in intensity to that of the background emission signal at the measured wavelength. The BECs for each emission line were determined by calibration without background correction, followed by a sample analysis with the shutter to the optics closed. The BECs obtained are displayed in Table 3, alongside reference values as stated by the instrument manufacturer.

BEC values were superior to instrument specifications, with the exception of the P 177.428 line. It is recognised that performance in the low wavelength UV region may be improved under conditions of high flow optics nitrogen purge settings, however, the instrument was typically operated under normal purge settings.

Multiple level calibration curves were generated for each spectral line of interest and subjected to linear least mean squares regression analysis, with calculated correlation coefficients, $r > 0.999$. The LDR for each line

Table 2
Instrument and method detection limits

Element	IDL (μg/l) [a]	IDL (μg/l) [b]	MDL (μg/100g) [c]
Ca	0.08	0.02	nd [d]
Cu	0.35	0.4	12
Fe	1.5	2	44
K	17	20	nd
Mg	0.08	0.07	nd
Mn	0.3	0.4	3
Na	2.7	3	nd
P	26	30	nd
Zn	1.2	1	68

[a] Measured IDLs, 3σ method, $n = 10$ (instrumental conditions as per Table 1(a)).

[b] Instrument specification IDLs (instrumental conditions as per Table 1(a)).

[c] Measured MDLs, $n = 10$, calculated as std dev $\times t$, where $t = 1.833$ from one-sided t-distribution at 95% confidence level (instrumental conditions as per Table 1(b)).

[d] nd = not determined.

Table 3
Background equivalent concentration (BEC) data (ppm)[a]

Emission line	Observed	Declared
Ca 422.673	0.15	0.33
Cu 324.754	0.08	0.18
Fe 259.940	0.10	0.21
K 766.491	3.07	7.1
Mg 279.553	0.004	0.01
Mn 257.610	0.02	0.05
Na 588.995	0.54	0.99
P 213.618	0.63	2.56
Zn 213.856	0.04	0.06

[a] Established under conditions as described in Table 1(a).

relative to its detection limit was estimated, and ranged from greater than 4 orders (Na, P) to almost 7 orders (Ca). This concurs with the generally accepted improved linearity of the ICP-OES technique relative to AAS (2–3 orders), and makes viable the simultaneous analysis of samples containing elements over a wide concentration range.

Instrument and method precision data is displayed in Table 4. Acceptable instrumental precision of the ICP-OES is demonstrated, while method precision ranged 0.5–2.5% for within-run repeatability and 0.9–2.5% for between-run reproducibility. An additional indication of method precision, the Horwitz ratio, while primarily intended for estimates of interlaboratory variability, is also useful in method validation (Boyer et al., 1985; Thompson & Lowthian, 1995; Albert & Horwitz, 1997) and ranged 0.1–0.4 for all elements, well below the acceptable limit of 0.5–2.0. $RSD_r : RSD_R$ values for all elements ranged 0.3–0.8, with the exception of Cu (1.0), and comply with accepted criteria for the relationship between repeatability and reproducibility precision (Albert & Horwitz, 1997).

Table 4
Instrument and method precision data

Element	RSD (%)[a]	RSD_r (%)[b]	RSD_R (%)[c]
Ca	0.19	0.5	1.2
Cu	0.19	2.5	2.5
Fe	0.27	0.5	1.0
K	0.22	0.7	0.9
Mg	0.2	0.5	1.2
Mn	0.17	0.7	1.8
Na	0.23	0.7	0.9
P	0.24	0.5	1.6
Zn	0.51	0.8	2.4

[a] Instrumental precision, $n = 10$ (estimated with reference to aqueous standards under conditions described in Table 1(a)).

[b] Within-run method precision, $n = 15$ (estimated with reference to in-house infant formula control powder under conditions described in Table 1(b)).

[c] Between-run method precision, $n = 15$ (estimated with reference to in-house infant formula control powder under conditions described in Table 1(b)).

Table 5
Analyte recovery (%)[a]

Element	[Level (%)]		
	50	100	150
Ca	100.9	99.2	99.4
Cu	100.2	101.1	100.1
Fe	88.9	100.5	101.5
K	101.3	99.8	100.0
Mg	99.2	97.3	97.4
Mn	99.6	99.4	97.9
Na	101.6	98.5	97.8
P	103.7	101.7	102.2
Zn	99.3	97.2	95.9

[a] Values are means of between-run duplicate analyses.

An estimation of analyte recovery was measured after spiking the in-house control sample at the 50, 100, and 150% levels with authentic elemental standards, and data listed in Table 5. Recoveries were essentially quantitative with the possible exception of Fe at the 50% level (88.9%). Three reference samples were used to determine the accuracy of the overall method. These included a well characterised in-house control sample, the NIST 1846 SRM certified infant formula (''non-certified'' values declared for mineral content) and the NIST 1549 SRM certified non-fat milkpowder, and the results are shown in Table 6.

Comparability of results based on independent methods is indicative of an unbiased estimate of the true analyte level. The data demonstrates both compliance for SRMs within declared confidence intervals and agreement with independent AAS and spectrophotometric techniques for the in-house control sample. Additional independent comparative data against both conventional dry-ashing and acid-digestion protocols was obtained for a range of infant formulas and milks with application of either AAS or ICP-OES and these results are listed in Table 7.

For the six infant formulas studied (A–F), the overall method means for each analyte were subjected to statistical analysis for variance and are graphically presented in Fig. 1. The graphs represent a test for method comparability for each individual element, with overlap of mean standard error least significant difference (LSD) limits indicating an absence of significant difference ($p = 0.05$).

In general, there was acceptable comparability between all four independent methods for each of the nine elements, although Cu appeared to be subject to increased variability, a consequence of its lower concentration and possible contamination during the ash procedure. There was no apparent systematic bias for the proposed direct slurry dispersion method, which was consistently equivalent to at least two of the three independent alternative methods. Slurry instability was

Table 6
Comparative data (mg/100g)

Element	In-house control ($n=15$)		NIST 1846 SRM ($n=15$)		NIST 1549 SRM ($n=8$)	
	ICP [a]	FAAS [a]	ICP [a]	Certified [b]	ICP [a]	Certified [b]
Ca	626 (8)	642 (13)	363 (10)	367 (20)	1230 (25)	1300 (50)
Cu	0.434 (0.010)	0.431 (0.011)	0.484 (0.018)	0.504 (0.027)	0.070 (6)	0.070 (0.010)
Fe	7.44 (0.08)	7.86 (0.20)	5.92 (0.12)	6.31 (0.40)	0.188 (0.009)	0.178 (0.010)
K	767 (7)	794 (17)	718 (19)	716 (38)	1620 (35)	1690 (30)
Mg	58.0 (0.7)	57.9 (1.0)	54.7 (1.3)	53.8 (2.9)	116 (3)	120 (3)
Mn	0.156 (0.003)	0.168 (0.006)	0.034	0.040 [c]	0.025 (0.004)	0.026 (0.006)
Na	221 (2)	223 (6)	220 (6)	231 (13)	463 (10)	497 (10)
P	427 (7)	429 (8)	257 (7)	261 (15)	1070 (25)	1060 (20)
Zn	3.64 (0.09)	3.80 (0.07)	5.90 (0.13)	6.00 (0.32)	4.48 (0.19)	4.61 (0.22)

[a] Mean values are shown, standard deviations in parantheses.
[b] NIST certified values are shown as mean, $2 \times$ CV in parantheses.
[c] Indicative value only.

occassionally observed for sample A, which would contribute to the calculated method variance.

Application of the described, validated method suggests that QC analysis of infant formulas, milk and milk powders may be significantly improved with respect to sample throughput and could, in principle, be applied to other similar food types. While the sample preparation scheme represents the principal benefit, other method attributes are also considered to be significant. Thus, the multiple benefits derived from the inherent simultaneity of OES as compared to AAS techniques, have been demonstrated in this study and confirm the recent findings of others as applied to various food types (Barnes & Debrah, 1997). For ICP-OES instruments incorporating a CTD detector, the ability to concurrently access multiple element lines provides further analytical confidence, while the potential to determine emission lines in the far UV (P, Al, S, and I) may eliminate the need for independent techniques for these analytes. LDRs over several orders minimise the requirement for multiple dilutions and measurements, further contributing to the overall reduction in analysis time. As reported previously (Miller-Ihli, 1996), instrumental operating parameters may be successfully configured under compromise conditions for all elements of interest, a feature facilitating an additional contribution to analytical throughput.

The direct introduction of intact milk dilutions into an IC plasma relies on the integration of sample destruction, atomisation and excitation, essentially within a single operation. Slurry nebulisation has received increased attention recently (de la Guardia et al., 1986; Ruiz et al., 1995; Sturup & Buchert, 1996; Bermejo et al., 1997; Ebdon et al., 1997; Nobrega et al., 1997), with several of its potential limitations eliminated or accommodated. There are several attributes of a slurry which have been acknowledged as critical to the stability, homogeneity, transport and nebulisation efficiency of the intact sample. These include particle size, slurry concentration, zeta potential and the use of dispersants, and have been thoroughly reviewed previously (Ebdon et al., 1997).

Provided a diluted milk slurry displays transport and atomisation efficiencies equivalent to those of aqueous standards, then a direct external calibration may be appropriate. However, in order to account for potential deviations from such ideal behaviour due to matrix effects, an alternative calibration method is advocated as a precautionary measure. While the standard additions technique is well accepted, we have selected the internal standard method through incorporation of Lu, which successfully compensated for matrix variations in transport and atomisation efficiencies between aqueous standards and milk samples for all elements of interest. This approach eliminated the requirement for matrix-matching while improving both accuracy and precision. Interestingly, a recent study reported the dispersion of non-fat milkpowders in a mixed tertiary amine solution which facilitated dissociation of the casein micelles, resulting in clear, colourless solutions that were therefore quantifiable directly against an external calibration (Nobrega et al., 1997).

A practical concern in direct dispersion nebulisation is the potential for system blockage by solid residue deposited from the sample or nebuliser streams. However, the recommended post-run clean-up procedure allowed for the analysis of up to 2000 samples before a cleaning and maintenance schedule of the sample introduction hardware is required. The cone-spray, high solids nebuliser performed satisfactorily and while alternative nebuliser types (cross-flow, Meinhard, etc.) may provide better performance with respect to sensitivity, their ability to tolerate high solids content is uncertain.

The method as described, has been successfully applied to a wide range of infant formulas and milks, both powdered and fluid. However, certain dry-blended

Table 7
Comparison of techniques (mg/100g)[a]

Element		[Sample[b]]							
		A	B	C	D	E	F	G[c]	H[d]
Ca	1	375	463	847	485	427	608	157	116
	2	391	468	882	507	378	584	157	118
	3	379	468	838	522	391	604	nd	nd
	4	367	435	846	492	370	593	nd	nd
Cu	1	0.199	0.394	0.450	0.354	0.404	0.480	(0.001)	(0.001)
	2	0.249	0.437	0.480	0.361	0.411	0.487	(0.009)	0.041
	3	0.178	0.373	0.410	0.332	0.379	0.451	nd	nd
	4	0.194	0.385	0.409	0.337	0.376	0.453	nd	nd
Fe	1	2.45	6.18	7.82	6.53	6.86	8.00	(0.03)	(0.02)
	2	2.79	6.27	7.89	6.29	6.77	7.57	(0.04)	0.05
	3	2.45	5.96	7.33	6.27	6.62	7.57	nd	nd
	4	2.55	6.14	7.57	6.53	7.02	8.09	nd	nd
K	1	521	743	1040	478	596	718	191	144
	2	520	740	1060	472	499	674	191	136
	3	532	760	1050	500	544	742	nd	nd
	4	564	769	1068	512	570	764	nd	nd
Mg	1	39.5	54.4	76.4	47.6	42.9	55.7	14.2	11.1
	2	40.2	54.3	77.2	47.0	36.6	52.6	14.1	11.1
	3	37.5	51.7	71.8	45.6	36.9	52.9	nd	nd
	4	40.1	54.1	75.5	45.1	38.1	54.3	nd	nd
Mn	1	0.404	0.086	0.114	0.074	0.087	0.109	0.004	(0.003)
	2	0.411	0.094	0.121	0.076	0.080	0.108	0.005	(0.003)
	3	0.380	0.081	0.107	0.072	0.080	0.104	nd	nd
	4	0.405	0.092	0.106	0.073	0.079	0.108	nd	nd
Na	1	197	230	270	215	215	219	50.9	41.2
	2	165	232	280	218	187	212	50.4	46.8
	3	166	235	277	228	209	231	nd	nd
	4	174	235	277	223	211	219	nd	nd
P	1	214	337	601	342	295	392	117	91.3
	2	218	324	587	352	249	357	116	89.6
	3	220	331	591	371	274	387	nd	nd
	4	231	335	581	366	279	392	nd	nd
Zn	1	1.37	3.48	3.90	3.65	4.15	4.23	0.46	0.35
	2	1.45	3.52	3.92	3.62	3.82	4.14	0.47	0.36
	3	1.56	3.59	4.20	3.74	3.95	4.49	nd	nd
	4	1.55	3.37	3.89	3.76	3.76	4.25	nd	nd

1: Slurry dispersion/ICP-OES test method.
2: Dry-ash sample preparation/ICP-OES method.
3: Wet digestion sample preparation/ICP-OES method (independent laboratory).
4: Wet digestion sample preparation/FAAS(independent laboratory).

[a] Results are means of independent, duplicate, between-day analyses.

[b] A: soy-based infant formula (IF), oil-filled; B: goatmilk IF; C: milk-based follow-on, partially oil-filled; D: milk-based IF, oil-filled; E: whey-based IF, oil-filled; F: whey-based IF, partially oil-filled.

[c] Liquid milk, 0.1% fat.

[d] Liquid milk, 3.3% fat nd: not determined. Values in parentheses indicate $<$ MDL.

infant formulations, soya-based formulas and products supplemented with calcium phosphate exhibited slurry instability, resulting in partial precipitation which affected Ca and Mg recoveries. Whether this is an intrinsic characteristic of these products or dependent on the individual formulation is unclear. However, preliminary trials incorporating the sample solubilisation protocol of Nobrega et al. (1997) suggest that improvements for such samples are achievable.

Finally, the description of diluted milk or reconstituted milk powder as a "slurry" may be ambiguous since as a colloidal entity it may be more accurately

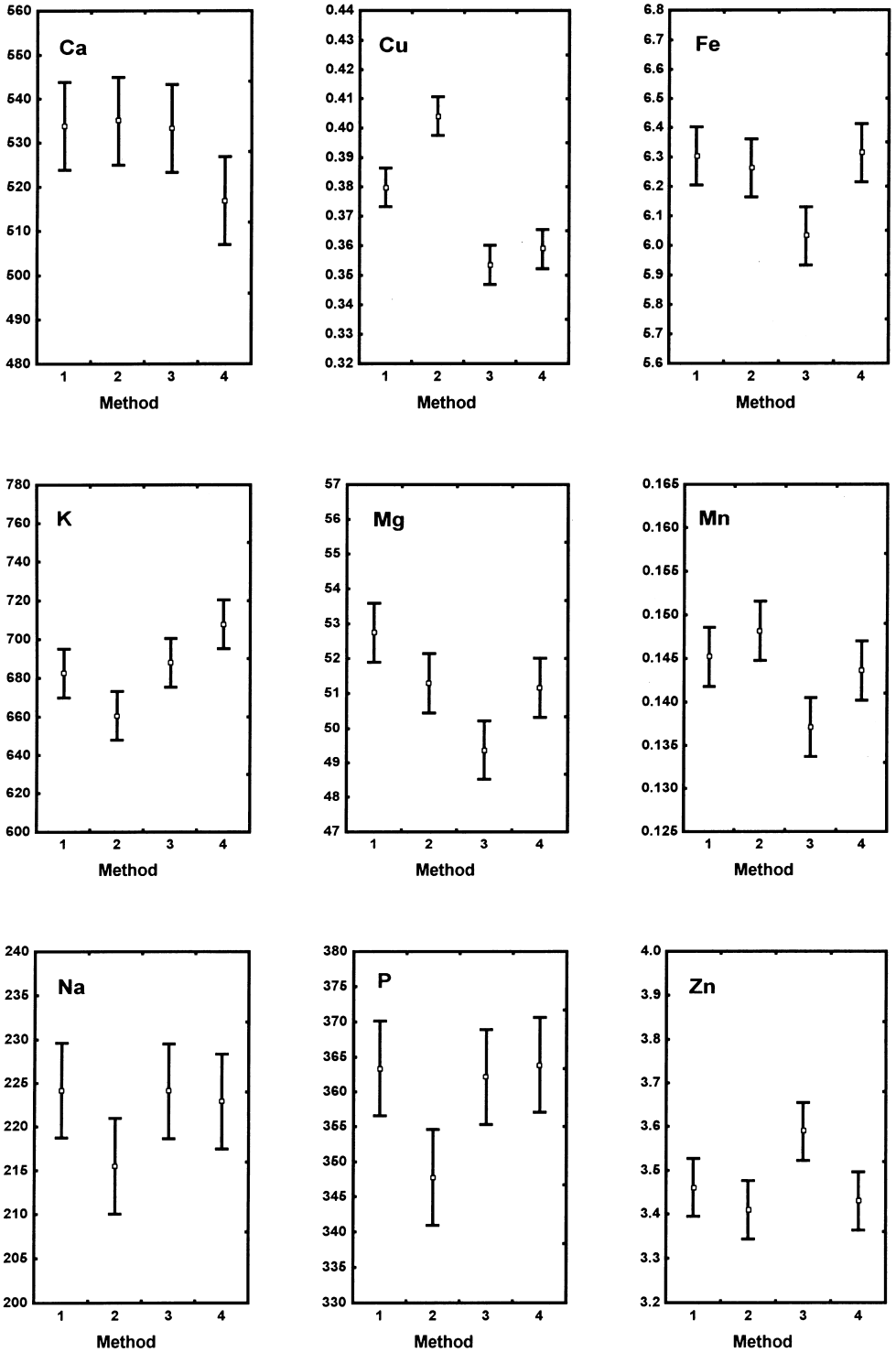

Fig. 1. Comparison of methods. Error bar represents the Least Significant Difference (LSD) limits derived from the standard error of the means ($p = 0.05$). Methods as described in Table 7. All units in mg/100g.

characterised as a solution. However, the several recent literature references to slurry nebulisation of milk suggests the acceptance of this terminology, though the term "direct dispersion" could be applied as a suitable alternative description.

4. Conclusion

The slurry nebulisation technique, in conjunction with a CTD-based ICP-OES instrument, provides a validated, rapid and robust methodology for the analysis of

the majority of milks and infant formulas, and has facilitated a significantly increased throughput in QC compliance monitoring of infant formula. The facile sample preparation scheme, true spectral simultaneity, compromise instrumental conditions, and multiple order LDRs all contribute to increased analytical throughput and represent significant advantages relative to traditional spectroscopic techniques as applied to dairy products.

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