

## Rapid Multimineral Determination in Infant Cereal Matrices Using Wavelength Dispersive X-ray Fluorescence

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A rapid and simple method for the determination of a series of macroelements (sodium, magnesium, phosphorus, chlorine, potassium, and calcium) and trace elements (manganese, iron, and zinc) by wavelength dispersive X-ray fluorescence has been developed and validated for infant cereal matrices. Reference values were obtained by inductively coupled plasma optical emission spectroscopy and by potentiometry. The 88 investigated samples were commercially available products. Pellets of 4 g were prepared under 10 tonnes of pressure. For each sample, 3 pellets were prepared and analyzed. Limits of quantification and repeatabilities were evaluated. Calibrations were established with 43 samples, and method validation was made using a second set of 45 samples. An evaluation of this alternative method was done by comparison with data obtained from the reference methods. The results show the good performances of the alternative method to routine infant cereals analysis.

**KEYWORDS:** WDXRF; infant cereals; food; ICP-AES; potentiometry; mineral; sodium; magnesium; phosphorus; chloride; potassium; calcium; manganese; iron; zinc

### INTRODUCTION

The addition of minerals (and/or vitamins), also called fortification, is a common way (1, 2) to allow the standardization of the nutrient content in foods (that naturally show variable concentrations) and thus to ensure the consistency of the product quality, to restore those nutrients lost during processing and then to maintain the nutritional quality of foods, to provide correction and prevention of nutritional deficiencies in the population, and to add value to finished products.

To ensure correct addition of premixes during infant formula or infant cereal production, analytes such as calcium, iron, or zinc may be used as tracers and thus can be determined in final products. Data obtained with accurate and rapid analyses can be used to adapt the process parameters and thus to ensure target concentration of added premixes is achieved. When target concentrations are obtained in compliance with the declaration, the production can be released.

In this connection, since the advent of greater control of starting raw materials as well as finished food products, the development of rapid, robust, multielemental, and accurate techniques to obtain data about the origin, identity, nutrition, or processing has become a priority. The major advantage of the X-ray fluorescence (XRF) technique compared to wet chemical analysis is that the measurements can be carried out directly on solid samples (powder in a sample cup or pressed into pellets). This avoids lengthy and laborious sample preparation steps, using corrosive and toxic reagents, which in turn

lead to contamination risks. Less manipulation and fewer time-consuming digestions mean cost and time savings. Further advantages of XRF include simplicity of use, short analysis time, and simultaneous analysis of elements, leading to a high throughput. The XRF technique possesses the main characteristics of an analytical tool to be used near the food production lines (3). For milk powders and infant formulas, the use of the X-ray fluorescence technique shows successful results (4, 5), thus minimizing risks of recall or rework.

In the present study, a wavelength dispersive (WDXRF) method is proposed for the rapid checking of major minerals in finished infant cereals. Calibrations were established using a suitable range of products of infant cereals. These “calibration products” contain a wide range of mineral values, which have been analyzed using reference methods.

### MATERIALS AND METHODS

**Trial Samples.** Infant cereal samples were worldwide commercially available. Two groups of samples were used during this study: the calibration set including 43 samples and the validation set including 45 samples.

Reference values of all samples are compiled in **Table 1**.

Prior to all analyses, infant cereal samples were homogenized using a coffee grinder. No synthetic sample was produced by spiking.

**Reference Methods.** Reference values of all samples were determined using in-house-validated procedures by ICP-AES for Na, Mg, P, K, Ca, Mn, Fe, and Zn and by potentiometry for chloride. Analyses were systematically made in duplicate.

A Varian Vista-AX Axial ICP-AES instrument (Varian, Mulgrave, Australia) equipped with a charge-coupled device (CCD) detector was

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**Table 1.** Reference Values (Milligrams per Kilogram) of the Samples and Identification of the Calibration Samples

sample	Na	Mg	P	Cl	K	Ca	Mn	Fe	Zn	calib <sup>a</sup>	sample	Na	Mg	P	Cl	K	Ca	Mn	Fe	Zn	calib <sup>a</sup>
1	2717	724	3640	2231	5502	5564	9.3	89.7	76.8	X	45	62	179	839	478	1174	290	5.5	91.7	4.4	
2	2666	774	3693	2192	5777	5472	10.0	88.4	75.6		46	62	398	1073	460	1968	352	7.1	113.7	6.9	
3	962	542	3249	3144	6731	5070	4.0	87.5	14.3		47	848	336	2512	1830	3897	4046	2.8	55.8	14.3	X
4	1155	425	3354	3126	5374	4069	3.3	76.5	14.1		48	398	178	973	893	2789	5931	3.6	61.2	6.5	X
5	921	494	3122	2865	6318	4729	3.7	77.5	13.1		49	163	244	1106	776	1579	331	8.0	100.5	6.1	
6	1319	494	4224	3605	6316	5858	2.8	84.9	17.7		50	109	250	874	389	1425	1052	5.9	90.7	8.6	
7	1261	463	3685	3331	5817	4563	3.9	82.3	24.0	X	51	43	393	554	29	2249	133	3.4	123.2	6.3	X
8	135	753	1748	549	4687	366	9.3	166.0	12.9		52	88	225	1819	695	3758	2527	6.4	132.2	5.3	
9	125	329	1261	714	1752	274	7.5	9.5	8.9		53	71	736	1779	<300	4087	394	10.9	16.6	15.7	
10	217	593	2051	712	3560	4818	9.0	202.5	9.1	X	54	68	547	1603	311	1709	314	10.9	12.0	14.4	X
11	210	547	2211	918	2979	4246	8.0	166.2	6.2	X	55	478	667	2896	1107	4664	5479	5.7	168.7	14.7	
12	1238	1037	3764	2500	6543	5757	23.9	98.6	40.8	X	56	394	446	2691	1437	4063	5178	3.3	143.4	10.4	X
13	1039	399	3083	3251	5027	3936	4.1	85.2	13.7	X	57	906	330	2371	2438	3746	4347	2.5	27.0	16.5	
14	135	405	2105	839	6106	2637	11.7	129.7	8.8		58	398	584	1965	1222	3924	2350	8.5	18.7	13.5	X
15	121	303	2320	653	3953	3192	9.1	220.6	9.8	X	59	415	512	1945	1114	2837	2407	8.5	19.5	14.6	
16	1172	543	3352	3808	7147	4085	4.0	82.7	14.5		60	1082	676	2161	1976	4152	2746	8.1	64.2	42.0	X
17	1349	492	3644	3767	5787	4543	7.7	69.4	18.0		61	1594	745	2422	2723	5069	3102	6.7	71.1	45.4	
18	1300	468	3476	3606	6593	4516	3.2	84.3	14.8	X	62	2024	908	3022	3667	3956	346	26.4	28.4	18.5	X
19	160	429	1459	574	2978	1766	6.9	209.7	8.9	X	63	45	178	830	<300	777	286	8.0	5.1	20.1	
20	1219	504	3375	3545	6668	4334	6.7	78.8	14.2		64	50	150	792	<300	760	696	6.1	6.1	18.4	X
21	227	396	1260	526	1561	1109	5.3	125.0	8.1	X	65	56	934	2965	471	4100	395	27.4	90.5	21.0	
22	1646	743	3742	2818	5709	4891	3.6	69.3	20.1		66	45	960	3053	572	3902	382	27.7	31.2	20.3	X
23	1732	620	3920	3684	6103	4919	2.7	68.6	18.9	X	67	96	853	2397	596	3957	362	20.5	24.5	15.1	X
24	1871	1393	2253	817	1473	424	12.5	110.4	13.7	X	68	125	815	2509	534	3687	564	19.8	77.2	15.8	
25	1443	1260	1943	353	1209	457	10.5	117.5	11.5	X	69	46	934	2844	441	3817	404	23.2	91.8	19.2	
26	235	318	1755	416	2373	1566	10.1	100.4	14.3		70	93	362	3309	737	1851	3343	8.8	87.8	8.4	X
27	1610	753	4135	4234	6498	5685	2.6	89.4	21.2	X	71	97	335	3493	692	1646	3472	7.6	96.2	7.8	
28	1411	528	3263	3812	6428	4331	2.9	73.8	16.3		72	106	527	3561	612	2217	3303	7.4	96.7	9.4	
29	1380	537	3426	3653	5517	4298	5.8	79.6	17.3	X	73	99	463	3475	383	1357	3138	7.0	77.9	10.5	
30	1993	693	3815	3380	6824	4462	3.9	81.7	19.1		74	158	310	1443	590	1539	3519	7.2	99.2	6.8	X
31	1098	978	4003	2796	5028	2543	16.3	160.1	25.2		75	103	304	1254	603	1422	3085	8.0	89.0	7.3	X
32	119	271	1341	810	2200	2535	6.5	153.6	7.0		76	60	188	793	676	1363	328	5.8	6.9	3.9	X
33	412	262	1428	1146	2054	2461	6.0	173.2	8.6	X	77	124	462	1073	560	3824	361	6.5	21.4	7.0	
34	359	272	1440	1068	2094	2984	6.4	110.8	7.4	X	78	2240	246	1845	3176	1695	2294	5.4	28.5	7.2	
35	1340	580	3719	3832	7529	4432	3.8	76.7	16.5	X	79	100	335	1158	739	1765	1859	8.4	137.1	7.3	X
36	1258	463	3359	3419	5874	3999	4.2	75.5	14.4		80	97	311	1126	673	1767	2010	8.4	150.1	6.8	X
37	1248	649	3254	3386	5634	4026	4.5	83.6	15.7		81	99	348	1214	679	1782	1920	8.6	148.5	7.1	
38	1275	505	3430	2871	6675	4413	5.2	75.7	13.3	X	82	171	396	1384	368	1086	1251	6.4	155.6	8.8	X
39	79	204	789	254	658	197	4.8	83.4	8.1		83	128	303	1095	390	902	1243	6.5	125.0	12.2	
40	201	212	1021	861	1522	309	6.9	98.5	5.2	X	84	146	413	1286	413	1187	1093	4.5	127.1	7.8	X
41	207	503	1338	1156	6632	1214	10.9	86.5	10.9	X	85	162	382	1234	427	1184	1168	4.5	123.7	7.6	
42	67	1199	3380	729	5556	449	23.9	31.9	23.3		86	96	385	1368	793	1871	1844	9.6	129.7	9.4	
43	119	1096	3307	544	3906	372	28.3	32.4	26.6	X	87	102	442	1397	864	1977	2444	8.2	191.3	8.9	
44	1521	526	4200	4103	6915	4678	4.3	43.2	17.3	X	88	97	392	1356	651	1766	2053	10.0	160.0	8.1	X

<sup>a</sup> Samples with "X" were used in the calibration set. The others were used in the validation set.

**Table 2.** Measurement Conditions Used in This Study

	radiation	crystal <sup>a</sup>	detector <sup>b</sup>	counting time (s)	kV	mA	collimator (deg)	empirical correction	2θ (deg)	internal standard
Na	Kα	LSM	SG	600	30	2	0.25	Lachance Traill		Rh Ka
Mg	Kα	LSM	SG	600	30	2	0.25	no		Rh Ka
P	Kα <sub>1,2</sub> background	PET	FPC	60	30	2	0.25	Lachance Traill	89.350	Rh Ka
		PET	FPC	60	30	2	0.25		93.450	Rh Ka
S	Kα <sub>1,2</sub> background	PET	FPC	60	30	2	0.25	no	75.712	Rh Ka
		PET	FPC	60	30	2	0.25		77.997	Rh Ka
Cl	Kα <sub>1,2</sub> background	PET	FPC	60	30	2	0.25	Lachance Traill	65.372	Rh Ka
		PET	FPC	60	30	2	0.25		67.263	Rh Ka
K	Kα <sub>1,2</sub> background	LiF200	FPC	60	30	2	0.25	Lachance Traill	136.637	Rh Ka
		LiF200	FPC	60	30	2	0.25		129.410	Rh Ka
Ca	Kα <sub>1,2</sub> background	LiF200	FPC	60	30	2	0.25	Lachance Traill	113.098	Rh Ka
Fe	Kα <sub>1,2</sub> background	LiF200	FPC	60	50	1	0.25	Lachance Traill	57.517	Rh Ka
		LiF200	FPC	60	50	1	0.25		61.620	Rh Ka
Zn	Kα <sub>1,2</sub> background	LiF200	SC	60	50	1	0.25	Lachance Traill	41.799	Rh Ka
		LiF200	SC	60	50	1	0.25		43.910	Rh Ka
Rh	Kα <sub>1,2</sub>	LiF200	SC	60	50	1	0.25	no	17.458	

<sup>a</sup> LSM, layered synthetic microstructure; PET, poly(ethylene terephthalate). <sup>b</sup> SG, sealed gas; FPC, flow proportional counter; SC, scintillator counter.

used. A cyclonic spray chamber (thermostated at 15 °C) coupled with a Micromist nebulizer (Glass Expansion, Melbourne, Australia) was used as the injection device, giving a signal of high precision. Chloride

quantifications were done using a titration unit consisting of a Titrimo DMS 716 C 730 sample changer C DM 141-SC electrode (from Metrohm) and following the silver nitrate titration method.

**Preparation of Samples for XRF Analysis.** Three pellets of 4 g of ground sample were pressed under 10 tons of pressure using a manual hydraulic press. The diameter of the pellet die was 32 mm. All accessories were delivered by Specac (Kent, U.K.). The pellets were solid enough to avoid use of a binder such as wax or cellulose. It was previously found that a constant weight has to be applied to only take into account analyte concentrations and matrix effect as factors of variability of XRF intensities.

**WDXRF Apparatus.** An ARL Optim'X from Thermo (Eublens, Switzerland) was used for this study with the following characteristics. **Table 2** gives the measurement conditions used for each analyte.

**Statistical definitions used for the analysis of calibration and validation data sets** are given below.

reference method value	$y_i$
WDXRF value	$\hat{y}_i$
no. of coefficients used in calibration equation	$p$
robust simple repeatability (used for WDXRF)	$SD_{rob}(r) = 1.1926 \times \text{Med}\{\text{Med}_{i,j=1,\dots,n,i \neq j}\{ x_i - x_j \}\}$
robust repeatability from duplicates (used for reference method)	$SD_{rob}(r) = 1.0484 \times \text{Med}_{i=1,\dots,n}\{ x_{i1} - x_{i2} \}$
robust repeatability limit	$r = 2.772[SD_{rob}(r)]$
difference	$d_i = \hat{y}_i - y_i$
bias	$\bar{d} = \frac{\sum_{i=1}^n (\hat{y}_i - y_i)}{n}$
standard error of calibration	$SEC = \sqrt{\frac{\sum_{i=1}^n (\hat{y}_i - y_i)^2}{n - p - 1}}$
difference standard deviation	$SD(d) = \sqrt{\frac{\sum_{i=1}^n (d_i - \bar{d})^2}{n - 1}}$
standard error of prediction	$SEP = \sqrt{\frac{\sum_{i=1}^n (\hat{y}_i - y_i)^2}{n}}$

## RESULTS AND DISCUSSION

**Limits of Quantification (LOQ) of the WDXRF Apparatus.** All samples were used for the determination of the LOQ. They have been defined as representing the lowest amounts of each analyte that can be determined with suitable accuracy and precision. They were established using two complementary criteria: recovery = average of the WDXRF three pellets results/reference value  $\times 100$  and relative standard deviation of the three pellets results measured by WDXRF.

The criteria conditions and LOQ are listed in **Table 3**.

**Repeatability of the WDXRF Apparatus.** Robust relative repeatability limits,  $r$  (%), at 95% confidence interval were calculated. Samples were chosen to cover the entire working range of the nine analytes and measured according to the following conditions: 12 pellets of each sample were prepared and analyzed by WDXRF the same day. Maximum relative limits are given in **Table 4** and are compared with ICP-AES reference method repeatabilities (calculated with duplicates on 20 samples during this study).

**Table 3.** Criteria Conditions and Limits of Quantification for Infant Cereals

analyte	range of recovery (%)	range of RSD of triplicates (%)	LOQ (mg/kg)
Na	90–110	0–5	500
Mg	90–110	0–5	300
P	80–120	0–5	500
Cl	90–110	0–5	300
K	90–110	0–5	500
Ca	90–110	0–5	600
Mn	80–120	0–10	6
Fe	80–120	0–10	30
Zn	80–120	0–10	10

**Table 4.** Robust Relative Repeatability Limits

analyte	$r$ (%) ICP-AES	$r$ (%) WDXRF
Na	<3	<6
Mg	<4	<3
P	<4	<2
Cl	<4	<3
K	<4	<4
Ca	<3	<2
Mn	<4	<16
Fe	<3	<7
Zn	<6	<4

**Table 5.** Calibration Characteristics for Different Elements

analyte	min (mg kg <sup>-1</sup> )	max (mg kg <sup>-1</sup> )	$n$	$R^2$	SEC (mg kg <sup>-1</sup> )
Na	848	2717	16	0.9199	137
Mg	303	1393	33	0.8605	84
P	554	4200	42	0.9702	196
Cl	311	4234	41	0.9925	120
K	760	7529	42	0.9940	160
Ca	696	5931	32	0.9868	186
Mn	10.0	28.3	10	0.9985	0.4
Fe	31.2	220.6	36	0.9620	9.7
Zn	10.4	76.8	22	0.9908	1.5

**Table 6.** Empirical Coefficients Used for the Calibrations

	$a_0$	$a_1$	$\alpha_P$	$\alpha_{Cl}$	$\alpha_K$	$\alpha_{Ca}$
Na	46.99	11643.79				
Mg	-93.68	25400.06				
P	-130.84	1553383.34		-1.801E-05		-1.769E-05
Cl	-31.51	3632.88				
K	132.32	30583.20				
Ca	141.32	30631.76				
Mn	1.14	7051.30			2.225E-05	2.703E-05
Fe	-51.11	357.21			4.643E-05	
Zn	2.62	79.98			1.111E-04	

Only  $r$  (%) values for concentrations above LOQ were taken into account. For Mn and Fe, XRF repeatability values are quite higher compared with the ICP-AES ones. This can be explained by their concentrations, which are quite lower compared to those of the other analytes, Na, Mg, P, K, and Ca, and closer to the LOQ of the WDXRF device.

**Calibration Characteristics.** Using the first set of samples, calibrations were established for Na, Mg, P, Cl, K, Ca, Mn, Fe, and Zn. Only concentrations above the LOQ (see **Table 3**) were taken into account. Both types of infant cereals (+ water and + milk) were included in the calibrations. The results are given in **Table 5**.

Different interelemental empirical corrections were applied to the following formula [Lachance Trill model (6)], with

**Table 7.** Summary of the Comparison between Reference and Alternative Methods<sup>a</sup>

analyte	min (mg kg <sup>-1</sup> )	max (mg kg <sup>-1</sup> )	n	R <sup>2</sup>	bias (mg kg <sup>-1</sup> )	model XRF = A + B(ref)		SD(d) (mg kg <sup>-1</sup> )	SEP (mg kg <sup>-1</sup> )	SEC (mg kg <sup>-1</sup> )	SEP/SEC (mg kg <sup>-1</sup> )
						A	B				
Na	906	2666	15	0.7481	-53	80	0.90	177	180	137	1.31
Mg	303	1199	29	0.6058	23	-19	1.08	62	65	84	0.77
P	789	4224	43	0.9363	-44	111	0.93	271	271	196	1.38
Cl	383	3812	38	0.9897	-14	29	0.96	138	141	120	1.18
K	658	7147	43	0.9903	-55	80	0.97	210	214	160	1.34
Ca	1052	5858	30	0.9779	-52	113	0.95	213	216	186	1.16
Mn	10.0	23.9	8	0.9671	0.2	2.0	0.88	1.2	1.1	0.4	3.14
Fe	47.0	191.3	36	0.9093	0.0	8.8	0.92	10.4	10.2	9.7	1.05
Zn	10.5	75.6	28	0.9892	0.0	0.2	0.99	1.3	1.3	1.5	0.87

<sup>a</sup> SEP, standard error of prediction; SD(d), difference standard deviation; R<sup>2</sup>, determination coefficient; A, intercept; B, slope.

**Table 8.** Comparison of Validation Performance Characteristics Obtained for Iron in Milk Powder and Infant Cereals

sample	analyte	min (mg kg <sup>-1</sup> )	max (mg kg <sup>-1</sup> )	n	R <sup>2</sup>	bias (mg kg <sup>-1</sup> )	model XRF = A + B(ref)		SD(d) (mg kg <sup>-1</sup> )	SEP (mg kg <sup>-1</sup> )	SEC (mg kg <sup>-1</sup> )
							A	B			
milk powders	Fe	35.9	97.9	26	0.9347	0.8	4.9	0.94	3.7	3.6	4.2
infant cereals	Fe	47.0	191.3	36	0.9093	0.0	8.8	0.92	10.4	10.2	9.7

**Table 9.** Specific Calibration Characteristics for Iron

sample	analyte	min (mg kg <sup>-1</sup> )	max (mg kg <sup>-1</sup> )	n	R <sup>2</sup>	SEC (mg kg <sup>-1</sup> )
all	Fe	31.2	220.6	36	0.9620	9.7
+ water	Fe	43.2	98.6	12	0.9230	4.9
+ milk	Fe	28.4	220.6	21	0.9525	11.9

C = concentration of analyte and I = WDXRF intensity. Empirical coefficients are given in **Table 6**.

$$C_{\text{Na}} = (a_0 + a_1 \times I_{\text{Na}})$$

$$C_{\text{Mg}} = (a_0 + a_1 \times I_{\text{Mg}})$$

$$C_{\text{P}} = (a_0 + a_1 \times I_{\text{P}})(1 + \alpha_{\text{Cl}} \times C_{\text{Cl}} + \alpha_{\text{Ca}} \times C_{\text{Ca}})$$

$$C_{\text{Cl}} = (a_0 + a_1 \times I_{\text{Cl}})$$

$$C_{\text{K}} = (a_0 + a_1 \times I_{\text{K}})$$

$$C_{\text{Ca}} = (a_0 + a_1 \times I_{\text{Ca}})$$

$$C_{\text{Mn}} = (a_0 + a_1 \times I_{\text{Mn}})(1 + \alpha_{\text{K}} \times C_{\text{K}} + \alpha_{\text{Ca}} \times C_{\text{Ca}})$$

$$C_{\text{Fe}} = (a_0 + a_1 \times I_{\text{Fe}}) \times (1 + \alpha_{\text{K}} \times C_{\text{K}})$$

$$C_{\text{Zn}} = (a_0 + a_1 \times I_{\text{Zn}}) \times (1 + \alpha_{\text{K}} \times C_{\text{K}})$$

**Global Validation.** The second set of samples (validation ones) was statistically evaluated using the calibrations developed above, only if the concentrations given by the reference methods were included in the calibration ranges or very close to the extrema. Statistical evaluation of the alternative method performance was performed using robust statistics (7) and the formulas described above. The results are given in **Table 7**.

For all of the analytes, A was found to be not significantly different from 0 and B was found to be not significantly different

from 1 for the model WDXRF = A + B (ref). This leads to the conclusion that results from the alternative method are similar to reference method results. For all of the elements the bias was found to be not significantly different from 0.

The ratio SEP/SEC is generally used as a final parameter for checking the validity of the method performance. With values lower than 1.5 for all of the elements (except Mn), their validation was found to be quite acceptable. The too narrow number of samples used for Mn validation could explain the high SEP value and SEP/SEC ratio.

The results of the validation are satisfactory and, except for iron, similar to those obtained in the previous study done on milk-based products (5). **Table 8** gives a comparison of the validation performance characteristics obtained for the infant formulas and infant cereals with the ARL Optim'X.

One can observe 2–3 times higher values for SD(d) and SEP obtained for infant cereals than those obtained for milk powders. It has been decided to split all of the previously studied infant cereals according to their preparation mode: addition of water or addition of milk.

**Specific Calibrations and Validations.** The performance characteristics of the calibration and validation processes are given in **Tables 9** and **10**. Without preparation indications (addition of water or liquid milk), 3 samples were not included in this second-step calculation.

After the sort according to families, the model XRF = A + B (ref) is still acceptable, with A not significantly different from 0 and B not significantly different from 1 for both kinds of samples. Nevertheless, a comparison of SEC, SEP, and SD(d) values shows the following trend:

“+ water samples” < all samples < “+ milk samples”

This can be explained, first, by the higher reference values for iron content found in samples requiring milk for preparation

**Table 10.** Specific Validation Characteristics for Iron

sample	analyte	min (mg kg <sup>-1</sup> )	max (mg kg <sup>-1</sup> )	n	R <sup>2</sup>	bias (mg kg <sup>-1</sup> )	model XRF = A + B(ref)		SD(d) (mg kg <sup>-1</sup> )	SEP (mg kg <sup>-1</sup> )	SEC (mg kg <sup>-1</sup> )
							A	B			
all	Fe	47.0	191.3	36	0.9093	0.4	8.8	0.92	10.4	10.2	9.7
+ water	Fe	47.0	88.4	13	0.8789	1.4	8.7	0.90	3.8	3.9	4.9
+ milk	Fe	77.2	191.3	20	0.8287	-3.6	-4.2	1.01	15.3	15.3	11.9

compared to samples requiring water for preparation. However, no difference in the production processes and in the form of Fe salts added could explain such differences.

Interestingly, only a small improvement in SD(d) and SEP values is observed when using specific calibrations.

**Conclusions.** The WDXRF technique shows good potential for quality control analyses of Na, Mg, P, Cl, K, Ca, Mn, Fe, and Zn in commercial infant cereals. Compared to EDXRF, the WDXRF technique is able to quantify sodium and magnesium in this type of matrix. Copper could not be analyzed due to insufficient detector sensitivity at the considered concentration level of <7 mg/kg.

Compared to reference methods, the WDXRF technique is quicker, with no sample preparation and no waste generation. Negligible analysis cost per sample, high throughput, and interesting payback time for investment in WDXRF equipment make this analytical technique very attractive for implementation in the food industry.

The key point for an efficient application is that a suitable range of products containing a large range of element contents must be available to obtain robust calibrations. The setting up of the calibration standards is an important step of the installation of this technique. Delays of several months are generally necessary to collect these calibration samples and to complete the reference measurements.

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