

Available online at www.sciencedirect.com



Food Chemistry

Food Chemistry 105 (2007) 280-285

www.elsevier.com/locate/foodchem

Analytical, Nutritional and Clinical Methods

Determination of trace element contents of baby foods from Turkey

Sibel Saracoglu^{a,*}, Kadriye O. Saygi^b, Ozgur D. Uluozlu^b, Mustafa Tuzen^b, Mustafa Soylak^c

^a Erciyes University, Faculty of Education, 38039 Kayseri, Turkey

^b Gaziosmanpasa University, Faculty of Science and Arts, Chemistry Department, 60250 Tokat, Turkey ^c Erciyes University, Faculty of Art and Science, Department of Chemistry, 38039 Kayseri, Turkey

Received 3 August 2006; received in revised form 25 September 2006; accepted 9 November 2006

Abstract

The levels of trace elements in different types of baby foods consumed in Turkey were determined by flame and graphite furnace atomic absorption spectrometry. Dry, wet and microwave digestion procedures were compared and the microwave digestion method was found to be the best. The accuracy of the method was ensured by using a standard reference material (NIST-SRM 8418 Wheat Gluten). The levels of elements in analyzed samples were found to be under legal limits. The range of the investigated elements were $0.52-4.38 \ \mu g/g$, $0.22-7.20 \ \mu g/g$, $1.02-67.5 \ \mu g/g$, $0.92-37.2 \ \mu g/g$, $0.12-0.32 \ \mu g/g$, $2.02-68.8 \ \mu g/kg$, $10.7-66.8 \ \mu g/kg$, $0.05-10.3 \ \mu g/g$, $2.67-25.4 \ \mu g/kg$ for Cu, Mn, Fe, Zn, Se, Cr, Al, Ni and Co, respectively.

Keywords: Trace elements; Baby foods; Atomic absorption spectrometry; Turkey

1. Introduction

Trace elements play an important role in human biology, because they are either inadequately synthesized or not synthesized in the body. Trace amounts of some metals, manganese, copper, zinc, for example, are essential micro nutrients and have a variety of biochemical functions in all living organisms. While these elements are essential, they can be toxic when taken in excess. In addition, some metals like lead, do not occur naturally in the body, and their presence, usually as a result of occupational or pollution-related exposure, is detrimental to health and children are more sensitive to these metals than adults (Ashraf, Seddigi, Abulkibash, & Khalid, 2006; Divrikli, Horzum, Soylak, & Elci, 2006; Soylak, Colak, & Turkoglu, 2006a; Soylak, Colak, Turkoglu, & Dogan, 2006b). Metals such as aluminium, cadmium and lead are found throughout the environment and are present in virtually all food at extremely low levels. All of these contaminants are more likely to affect bottle-fed infants. Generally, infants fed formula made with tap water are at the highest risk from metals contaminating the water supply (NRDC, 2005; Soylak et al., 2006a; Soylak et al., 2006b). The European Union banned the use of cadmium in materials and components of vehicles put on the market after 1 July, 2003, and in new electrical and electronic equipment after 1 July, 2006. Some countries have also had success reducing pollution from incinerators, power plants and factories, thereby reducing emissions of mercury and other heavy metals at trace levels (Official Journal of the European Union, 2003).

Indeed some infant foods, such as commercial infant formulae, are deliberately fortified with essential elements such as zinc and copper to ensure that they provide infants' nutritional requirements for trace elements. Another important factor that must be borne in mind when assessing the exposure of infants to trace elements and

^{*} Corresponding author. Tel.: +90 352 4374933; fax: +90 352 4378834. *E-mail address:* saracs@erciyes.edu.tr (S. Saracoglu).

heavy metals in their diets is that because infants grow and develop very rapidly in their first year of life, their energy requirements and hence their food consumption is on average much higher relative to their body weight than that of adults and older children (FSA, 1999).

Multi-element surveys of baby foods have been published because of growing interest in trace element concentrations in infant foods. Rodriguez, Alaejos, and Romero (2000) determined concentrations of iron, copper and zinc in 56 samples of nature human milk from Canarian women and five samples of powdered infant formula. Cu, Mn, Zn, and Fe were determined in the digestion solutions (Yaman & Cokol, 2004). A statistical study of correlation, factorial, and discriminant analysis on the metal composition of different types of baby foods was carried out to establish the relationships between the metal concentrations and, therefore, differentiate the samples according to the type of milk by Rodriguez, Alaejos, and Romero (1999). Tripathi, Raghunath, Sastry, and Krishnamoorthy (1999) investigated concentrations of the essential elements Zn, Cu and potentially toxic elements Pb and Cd in different milk samples and baby food materials. Yebra, Moreno-Cid, Cespón, and Cancela (2004) have utilized direct measurement by flame atomic absorption spectrometry for the determination of traces of iron in milk powder and infant formulas. Seven different infant formulas and 18 different types of infant foods representative of infant diets in Italy were analyzed, with particular reference to Al and Zn intake by Plessi, Bertelli, and Monzani (1997).

Flame/graphite furnace atomic absorption spectrometry is the main instrument used for the determination of trace heavy metal contents in food analysis laboratories. Many analytical methods including AAS for trace element determination in food materials require decomposition of the sample. The wet and dry ashing and microwave digestion are used for sample decomposition prior to the determination of trace elements.

The purpose of the present study was to determine the concentration levels of some elements at trace levels in different types of baby foods consumed in Turkey. The performance of digestion procedures including dry ashing, wet ashing and microwave digestion was compared in the presented work.

2. Materials and method

2.1. Apparatus

A Perkin–Elmer Analyst 700 model atomic absorption spectrometer equipped with a deuterium background corrector was used for determination of trace elements. Measurements of Fe, Cu, Mn and Zn were carried out in an air/ acetylene flame. The concentrations of Se, Cr, Al, Ni and Co were determined with a graphite furnace atomic absorption spectrometer. Argon as inert gas was used in the study with the graphite furnace. Standard reference materials were digested in a Mileston Ethos D model closed system microwave oven (maximum temperature $300 \,^{\circ}$ C, maximum pressure 1450 psi). Teflon reaction vessels were used for all the digestion procedures. The reaction vessels were cleaned using 5 ml of concentrated nitric acid before each digestion.

2.2. Reagents

Analytical reagent-grade chemicals were employed for the preparation of all solutions. Doubly distilled deionised water (Milli-Q Millipore 18.2 M Ω /cm) was used in all experiments. HNO₃ and H₂O₂ were of suprapure quality (E. Merck). All the plastic and glassware were cleaned by soaking in dilute nitric acid (1 + 9) and were rinsed with distilled water prior to use. The standard solutions of analytes for calibration procedure were produced by diluting a stock solution of 1000 mg/l of the all the investigated elements supplied by Sigma.

2.3. Sample collection

Nineteen different infant formulas from six different manufacturers in Kayseri, Turkey were purchased from the local markets and taken from Erciyes University Faculty of Medicine. Baby food samples were dried at a temperature of 105 °C for 24 h. The dried samples were homogenized using an agate homogenizer and stored in polyethylene bottles until analysed.

2.4. Digestion procedures

2.4.1. Dry ashing

The reliability of the method for estimation of trace elements in samples has been checked by analysing standard reference material (SRM). For digestion with dry ash, 0.2 g from standard reference material and 1 g from real samples was used. Samples were dry-ashed in furnace at a temperature of 450–500 °C and time of 16 h. The ashed sample was then treated with 1 ml of concentrated nitric acid for ash whitening and this mixture was ashed again for 6 h. Then, the residue was dissolved in 1–2 ml of concentrated HNO₃ and filtered through blue band filter paper. The sample was diluted to 10 ml with distilled water.

2.4.2. Wet ashing

Two-hundred milligrams from standard reference material and 1 g from real samples was used. Wet digestion of infant formula samples was performed using a mixture of $HNO_3:H_2O_2$ (6:2) (16.0 ml for a 1.0 g sample). This mixture was heated up to 130 °C for 4 h on the hot plate. Acid mixture was added again. After cooling, 5 ml of distilled water was added to the sample and mixed. The residue was filtered through blue band filter paper. Then the sample was diluted to 10 ml with distilled water. Blank digestions were also carried out in the same way.

2.4.3. Microwave digestion

For microwave digestion, the standard reference materials and baby food samples were accurately weighed around 0.20 g and 1.00 g, respectively. The samples were transferred to 100 ml teflon vessels. Samples were digested with 6 ml of HNO₃ (Suprapure, Merck), 2 ml of H₂O₂ (Suprapure, Merck) in a microwave digestion system and diluted to 10 ml with double deionized water (Milli-Q Millipore 18.2 MΩ/cm conductivity). Blank digestion was carried out in the same way. Digestion conditions for the microwave system were applied as 2 min for 250 W, 2 min for 0 W, 6 min for 250 W, 5 min for 400 W, 8 min for 550 W, vent: 8 min.

3. Results and discussion

Performance of digestion procedures using dry ashing, wet ashing and microwave digestion prior to the determination with AAS of some trace elements in baby food samples was compared. For that purpose, a **B5** baby formula sample was used and the results are given in Table 1. The slightly high trace metal levels were obtained when the microwave oven was used. The approximate time required for dry, wet and microwave digestions were 22 h, 4 h and 31 min, respectively. In light of these results, the microwave digestion procedure was chosen for the digestion of all the baby food samples, because of shorter required time and higher recovery (especially for Se).

The accuracy of results was verified by analyzing the concentration of copper, manganese, iron, zinc, selenium, chromium, aluminium, nickel and cobalt in NIST-SRM 8418 Wheat Gluten standard reference material. The certified and observed values for the SRM are given in Table 2. The results found by dry, wet and microwave digestion procedures except for Se with dry and wet digestion procedure are in good agreement with the certified values for the investigated analyte ions. The relative standard deviations were less than 10% for all the investigated elements. If the concentration levels of the most common matrix constituents of the reference standard materials analyzed and the accuracy of the presented methods are considered together, it can be concluded that the proposed method is free from interferences of the various constituents.

The detection limit based on three times the standard deviations of the reagent blank and characteristic mass based on 0.0044 absorbance were calculated for investigated analyte ions. The detection limit values of the

investigated elements for flame AAS were found to be 0.013 mg/l for Cu, 0.019 mg/l for Zn, 0.011 mg/l for Fe, 0.010 mg/l for Mn. The characteristic mass values were Al: 17 pg, Se: 22 pg, Co: 8 pg, Ni: 15 pg and Cr: 5 pg in graphite furnace AAS.

Trace metal levels in the 19 different infant formula samples analyzed by AAS after digestion by closed microwave system are given in Table 3. In the precision test, the average RSD% for all analytes were in the range of 1-10% (n = 18) for method. The results, which were repeated in triplicate, are given in Table 3.

The lowest and highest contents of copper were found as $0.52 \mu g/g$ for A5 and $4.38 \mu g/g$ for B5, respectively. The lowest and highest levels of zinc were found as $0.92 \,\mu g/g$ for **B1** and $37.2 \,\mu\text{g/g}$ for **E2**, respectively. The mean concentrations of Cu and Zn in investigated samples were 2.34 µg/g and 21.08 µg/g, respectively. Cu and Zn concentrations in **B2** baby formula sample used for low birth weight babies were higher than average Cu and Zn levels. It is known that Cu and Zn are nutrients and are essential for health. Copper and zinc contents of New Zealand infant formula have been reported in the range of $1.99-4.80 \ \mu g/g$ and $13.7-42.3 \ \mu g/g$, respectively. The concentrations of copper and zinc in the present study were therefore similar to those found in New Zealand (McKinstry, Indyk, & Kim, 1999). Copper and zinc values in baby food of India have been reported in the range of 1.11-3.16 µg/g and 9.37–34.59 µg/g, respectively (Tripathi et al., 1999). The mean concentrations of copper and zinc were 1.4 mg/kg (range less than 0.003 mg/kg to 5.3 mg/ kg) and 16.4 mg/kg (range of less than 0.06 mg/kg to 68.1 mg/kg), respectively in a MAFF study (FSA, 1999). Our results are consistent with mean of copper and zinc concentration of baby food in this MAFF survey and copper and zinc values in Indian baby food. The content of zinc in powder Italian infant formulas has been reported in the range of 13–110 μ g/g (Plessi et al., 1997). Our mean values for zinc were lower than mean value of zinc in Italy. The maximum tolerable daily intake of Zn is 0.3-1 mg/kg. Our values for Zn for all the samples were below WHO's values (WHO, 1982).

The lowest Mn level was found as $0.22 \ \mu g/g$ for **B1** and **C1** baby food samples, whereas the highest manganese level was 7.20 $\mu g/g$ in **F2**. The contents of manganese in some baby formulas were found in the range of 0.29–10.5 mg/kg in one study in Turkey (Yaman & Cokol, 2004). The range of manganese concentrations in our

Table 1

Trace metal contents with dry ashing, wet ashing and microwave methods in **B5** baby formula sample (n = 3)

Method	Concentrations $(\mu g/g)$									
	Cu	Mn	Fe	Zn	Se	Cr ^a	Al ^a	Ni	Co ^a	
Microwave	4.38 ± 0.31	3.01 ± 0.20	34.2 ± 2.8	32.4 ± 3.1	0.13 ± 0.01	29.2 ± 2.2	11.4 ± 0.7	4.98 ± 0.41	12.0 ± 1.1	
Wet ashing	4.10 ± 0.30	2.96 ± 0.25	32.1 ± 2.9	31.6 ± 2.9	0.10 ± 0.01	27.7 ± 2.5	10.7 ± 0.8	4.63 ± 0.42	11.8 ± 1.1	
Dry ashing	3.97 ± 0.36	2.90 ± 0.28	33.2 ± 3.2	31.2 ± 2.5	0.04 ± 0.01	26.5 ± 2.4	10.9 ± 0.9	4.21 ± 0.40	11.5 ± 1.2	

^a Cr, Al, Co (µg/kg).

S. Saracoglu et al. | Food Chemistry 105 (2007) 280-285

Results of the analysis with dry, wet and microwave procedure of NIST-SRM 8418 Wheat Gluten standard reference material ($\mu g/g$), $n = 3$									
Element	Certified value	Dry ashing	% Recovery	Wet ashing	% Recovery	Microwave	% Recovery		
Cu	5.94	$5.35\pm0.47^{\rm a}$	90	5.58 ± 0.30	94	5.87 ± 0.25	99		
Mn	14.3	13.6 ± 1.1	95	13.8 ± 1.2	97	14.1 ± 0.8	99		
Fe	54.3	52.1 ± 4.5	96	52.7 ± 3.2	97	53.8 ± 2.6	99		
Zn	53.8	49.5 ± 4.1	92	50.3 ± 3.3	93	52.5 ± 2.4	98		
Se	2.58	0.85 ± 0.07	33	2.19 ± 0.20	85	2.45 ± 0.21	95		
Cr	0.053	0.048 ± 0.005	91	0.049 ± 0.005	92	0.051 ± 0.004	96		
Al	10.8	9.60 ± 0.85	89	9.75 ± 0.65	90	10.3 ± 0.8	95		
Ni	0.13	0.12 ± 0.01	92	0.12 ± 0.01	92	0.13 ± 0.01	100		

0.01 ^a Mean \pm Standard deviation. 0.009 ± 0.001

90

Table 3

Co

Table 2

Trace metal contents ($\mu g/g$) in infant formula samples determined by AAS after digestion using the microwave digestion method (Mean \pm Standard deviation), n = 3

 0.009 ± 0.001

90

 0.010 ± 0.001

100

Code	Sample information	Cu	Mn	Fe	Zn	Se	Cr ^a	Al ^a	Ni	Co ^a
A1	For 4 month and onword baby, with added iron and prebiotic	1.97 ± 0.12	0.31 ± 0.02	11.0 ± 0.9	27.7 ± 1.8	0.12 ± 0.01	53.5 ± 4.3	17.7 ± 1.5	0.05 ± 0.01	2.69 ± 0.17
B 1	For 0–4 month, bottle formula	0.94 ± 0.10	0.22 ± 0.02	1.62 ± 0.12	0.92 ± 0.10	0.23 ± 0.02	9.70 ± 0.74	12.3 ± 0.9	0.07 ± 0.01	8.77 ± 0.43
B2	For premature, low birth weight baby	4.22 ± 0.28	0.30 ± 0.03	10.5 ± 0.8	28.0 ± 2.5	0.25 ± 0.02	20.2 ± 1.6	38.8 ± 2.4	0.05 ± 0.01	4.82 ± 0.31
C1	For newborn infant	2.63 ± 0.20	0.22 ± 0.02	33.7 ± 2.1	21.9 ± 1.6	0.15 ± 0.01	23.3 ± 2.1	30.8 ± 2.1	0.12 ± 0.01	10.1 ± 0.9
A2	_	3.74 ± 0.32	3.15 ± 0.20	42.5 ± 3.4	25.1 ± 2.1	0.26 ± 0.02	16.4 ± 1.5	31.2 ± 2.7	0.05 ± 0.01	21.4 ± 1.7
D1	With added iron, milk based, follow-on formula for 6 month onwards baby	4.17 ± 0.19	1.65 ± 0.12	31.5 ± 2.7	28.0 ± 1.9	0.24 ± 0.02	2.02 ± 0.14	13.4 ± 1.3	0.07 ± 0.01	2.67 ± 0.13
D2	Diarrhoea preventive	2.27 ± 0.16	0.30 ± 0.02	16.3 ± 1.5	19.1 ± 1.5	0.32 ± 0.03	23.8 ± 1.7	21.6 ± 1.8	4.21 ± 0.32	5.26 ± 0.37
B3	Vomit preventive	2.36 ± 0.22	1.61 ± 0.15	1.87 ± 0.16	20.3 ± 1.3	0.27 ± 0.02	23.3 ± 2.1	12.7 ± 1.1	4.38 ± 0.37	9.56 ± 0.70
B4	Special diet formula	1.59 ± 0.13	2.01 ± 0.14	11.4 ± 1.1	12.6 ± 1.2	0.25 ± 0.03	45.2 ± 3.5	24.5 ± 2.2	0.12 ± 0.01	5.87 ± 0.45
D3	Iron fortified first formula	1.48 ± 0.10	2.09 ± 0.12	50.2 ± 3.8	29.8 ± 2.7	0.19 ± 0.02	15.5 ± 1.4	36.2 ± 2.9	0.11 ± 0.01	10.9 ± 0.8
A3	_	3.36 ± 0.25	0.69 ± 0.07	44.2 ± 3.7	29.3 ± 2.1	0.15 ± 0.01	22.4 ± 1.5	35.9 ± 3.4	0.07 ± 0.01	14.0 ± 1.2
A4	With milk protein, 90%	1.69 ± 0.13	2.36 ± 0.21	17.2 ± 1.4	25.8 ± 2.3	0.12 ± 0.01	68.8 ± 5.2	66.8 ± 5.5	10.3 ± 0.9	8.70 ± 0.42
A5	_	0.52 ± 0.05	2.10 ± 0.17	1.02 ± 0.10	1.26 ± 0.10	0.18 ± 0.02	17.4 ± 1.4	10.7 ± 0.7	0.13 ± 0.01	10.8 ± 0.9
E1	Iron fortified formula, with milk, cereal and fruited, spoon formula	1.58 ± 0.14	3.29 ± 0.26	63.4 ± 5.1	13.0 ± 1.2	0.13 ± 0.01	13.4±1.1	24.5 ± 1.4	3.75 ± 0.24	25.4 ± 2.3
F1	With milk and mixed vegetable	1.71 ± 0.15	1.36 ± 0.12	33.2 ± 2.7	11.5 ± 1.1	0.16 ± 0.01	17.4 ± 1.4	16.4 ± 1.3	3.75 ± 0.19	22.5 ± 2.1
F2	Fruited	0.94 ± 0.10	7.20 ± 0.52	60.5 ± 5.5	7.28 ± 0.60	0.16 ± 0.02	13.2 ± 1.2	19.7 ± 1.6	0.10 ± 0.01	18.4 ± 1.6
E2	With milk and banana	1.32 ± 0.12	4.44 ± 0.35	67.5 ± 4.6	37.2 ± 2.6	0.16 ± 0.01	28.3 ± 2.6	27.4 ± 2.5	2.97 ± 0.18	8.87 ± 0.50
B5	For baby with allergic constitution	4.38 ± 0.31	3.01 ± 0.20	34.2 ± 2.8	32.4 ± 3.1	0.13 ± 0.01	29.2 ± 2.2	11.4 ± 0.7	4.98 ± 0.41	12.0 ± 1.1
B 6	_	3.56 ± 0.28	1.58 ± 0.13	53.3 ± 3.9	29.4 ± 1.7	0.12 ± 0.01	6.20 ± 5.7	24.2 ± 1.4	0.08 ± 0.01	10.3 ± 0.9

^a Cr, Al, Co (µg/kg).

samples is consistent with these results. In addition, manganese concentrations in infant formula in our study are similar to the range of 0.74 μ g/g to 4.04 μ g/g reported for formula tested in a study in New Zealand (McKinstry et al., 1999). While there is no recommended dietary allowance, the National Research Council's "estimated safe and adequate daily dietary intake" is 2-5 mg. The Institute of Medicine recommends that intake of manganese from food, water and dietary supplements should not exceed the tolerable daily upper limit of 11 mg per day (National Research Council, 1989). The intake of Mn with our investigated samples is well below this limit.

The concentration of iron in baby food samples was found in the range of $1.02-67.5 \,\mu g/g$. The contents of iron of some infant formula samples analyzed such as D3, E1, D1 were higher than others, because of added iron. The levels of manganese and iron were higher in specially fortified baby formula, for example E1, F2, E2. Iron value in infant formula of Spain has been reported in the range of 41.4-97.4 μ g/g (Yebra et al., 2004). The content of iron in some

Table 4	
Correlation coefficient between metal concentrations in baby for	bd

	Cu	Mn	Fe	Zn	Se	Cr	Al	Ni	Cc
Cu	1								
Mn	-0.265	1							
Fe	0.068	0.608	1						
Zn	0.631	-0.066	0.363	1					
Se	0.119	-0.242	-0.443	-0.182	1				
Cr	-0.180	-0.083	-0.323	0.202	-0.240	1			
Al	0.062	-0.016	0.147	0.383	-0.199	0.136	1		
Ni	-0.084	0.111	-0.066	0.137	-0.167	0.537	0.393	1	
Co	-0.156	0.471	0.541	-0.286	-0.283	-0.310	-0.016	0.102	1

r: 0.05.

powdered milk and cow milk samples was found in the range of 7.9–13.5 mg/l and 1.58–3.27 mg/l in a study in Portugal (Lima, Delerue-Matos, & Vaz, 1998).

The lowest and highest levels of selenium were found as $0.12 \ \mu g/g$ for A1, A4, B6 samples and $0.32 \ \mu g/g$ for D2, respectively. The concentrations of selenium in baby food of Spain have been reported in the range of 21.5– $72.8 \ ng/g$ (Vinas, Martnez, & Cordoba, 2000). Our values for Se were higher than value of selenium in Spain. Se is an essential element for nutrition. Selenium is an interesting trace element because it has an important antioxidant function, but if intake is excessive harmful effects appear, the difference between the necessary intake and that producing toxicity being small. It has been found that the selenium concentration in the range 2–8 mg/g in foods are harmful (Vinas et al., 2000).

The chromium level in the baby food samples varied from $2.02 \ \mu g/kg$ in **D1** to $68.8 \ \mu g/kg$ in **A4**. The chromium concentrations in investigated samples have been reported in the range of 0.01 mg/kg to 0.7 mg/kg in a MAFF survey. The mean Cr level was $0.12 \ mg/kg$ (FSA, 1999). The contents of chromium in our samples analyzed were lower than this survey. The mean levels of chromium of some milk based powder formula in Nigeria, UK and USA have been reported as 0.006, 0.005, 0.007 \ \mu g/ml in a study in USA, respectively (Ikema, Nwankwoalab, Odueyungboa, Nyavora, & Egiebora, 2002).

The lowest and highest contents of aluminium were found as 10.7 µg/kg for A5 and 66.8 µg/kg for A4, respectively. The mean of Al concentrations in baby food sample analyzed was 25.1 μ g/kg. Aluminium was detected in most samples with a mean concentration in all samples of 3.0 mg/kg (range less than 0.04 mg/kg to 28 mg/kg) in the archive published by MAFF (FSA, 1999). Aluminium value in powder infant formula of Italy has been reported in the range of $3-17 \,\mu\text{g/g}$ (Plessi et al., 1997). A tolerable daily intake (TDI) for aluminum of 1 milligram per kilogram of body weight per day has been established by an international committee of experts under the auspices of the World Health Organization (WHO) and the Food and Agricultural Organization of the United Nations (Health Canada, 2004). Even at the highest level report, intake of aluminum is well below the tolerable daily intake.

The lowest and highest contents of nickel were found as $0.05 \ \mu g/g$ for A1, B2, A2 and $10.3 \ \mu g/g$ for A4, respectively. The nickel concentrations were found in the range of $0.02 \ m g/kg$ and $0.21 \ m g/kg$ in 15 samples of formulae and baby foods (but not rusks) analyzed on a ready-to-feed basis in a MAFF survey of various foods (Ministry of Agriculture, Fisheries & Food, 1998). Cobalt levels in the samples varied from 2.67 $\mu g/kg$ in D1 to 25.4 $\mu g/kg$ in E1. The concentrations of copper, zinc, iron and selenium in our samples analyzed were below the nutrition reference value in Turkish Food Codex Regulations (Official Gazette of Turkey, 2000).

The correlation test was performed to investigate correlations between the metal contents in examined infant formula samples. The entire data were subjected to a statistical analysis and correlation matrices were produced to examine the inter-relationship between the investigated metal concentrations. The values of correlation coefficient between metal concentrations are shown in Table 4. The correlations between Fe–Mn, Zn–Cu, Co–Fe, Ni–Cr, Co–Mn are significant at the 0.05 level. The correlation coefficients (r values) for Fe–Mn, Zn–Cu, Co–Fe, Ni–Cr, Co–Mn were 0.608, 0.631, 0.541, 0.537, 0.471, respectively. The correlations between Zn–Al, Zn–Fe, Al–Ni, Cr–Fe, Cr–Co, Fe–Se are middle level. Se is correlated weakly and negatively between chromium, aluminium, nickel, manganese, zinc and cobalt.

4. Conclusions

The experimental results for the reference standard material (NIST-SRM 8418 Wheat Gluten) for dry, wet and microwave digestions except for Se with dry and wet digestion procedure were in agreement with the certified values. The proposed digestion methods were precise and accurate. The advantages of the present method are its simplicity, low cost, high speed of sample attack and rapid calibration. The microwave digestion procedure was the best because of more accuracy with respect to both time and recovery than dry and wet digestion. The analytical parameters obtained make this method suitable for the determination of Cu, Mn, Fe, Zn, Se, Cr, Al, Ni and Co in various baby foods. The levels of elements in analyzed samples were found to be under legal limits.

Acknowledgements

The authors are also grateful for the financial support of the Unit of the Scientific Research Projects of Gaziosmanpasa University and the Unit of the Scientific Research Projects of Erciyes University.

References

- Ashraf, W., Seddigi, Z., Abulkibash, A., & Khalid, M. (2006). Levels of selected metals in canned fish consumed in kingdom of Saudi Arabia. *Environmental Monitoring and Assessment*, 117, 271–279.
- Divrikli, U., Horzum, N., Soylak, M., & Elci, L. (2006). Heavy metal contents of some spices and herbal plants from western Anatolia to Turkey. *International Journal of Food Science and Technology*, 41, 712–716.
- FSA. (1999). Food standards agency, MAFF UK Metals and other elements in infant foods. Joint food safety and standards groups. Food surveillance information sheet number 190.
- Health Canada. (2004). Trace metal analysis infant formula. http:// www.hc-sc.gc.ca/fn-an/surveill/other-autre/infant-nourisson/ index e.html.
- Ikema, A., Nwankwoalab, A., Odueyungboa, S., Nyavora, K., & Egiebora, N. (2002). Levels of 26 elements in infant formula from USA, UK, and Nigeria by microwave digestion and ICP-OES. *Food Chemistry*, 77, 439–447.
- Lima, J. L. F. C., Delerue-Matos, C., & Vaz, M. C. V. F. (1998). Automation of iron and copper determination in milks using FIA systems and colorimetric detection. *Food Chemistry*, 62, 117–121.
- McKinstry, P. J., Indyk, H. E., & Kim, N. D. (1999). The determination of major and minor elements in milk and infant formula by slurry nebulisation and inductively coupled plasma – optical emission spectrometry (ICP-OES). *Food Chemistry*, 65, 245–252.
- Ministry of agriculture, Fisheries and food (1998). Cadmium, mercury and other metals in food. Food surveillance Paper No. 53. London: Stationery Office.
- National Research Council (1989). *Recommended Dietary Allowances* (10th ed.). Washington, DC: National Academy Press.
- NRDC. (2005). Natural resources defence council. Healthy milk, healthy baby chemical pollution and mother's milk. http://www.nrdc.org/ breastmilk/formula.asp.

- Official Gazette of Turkey. (2000). Turk food codex regulation, (Bulletin Number 2000/30), Issue: 24142.
- Official Journal of the European Union. (2003). Directive EC of the European parliament and of the council, 34, May 26.
- Plessi, M., Bertelli, D., & Monzani, A. (1997). Determination of aluminum and zinc in infant formulas and infant foods. *Journal of Food Composition and Analysis*, 10, 36–42.
- Rodriguez, E. M. R., Alaejos, M. S., & Romero, C. D. (1999). Chemometric studies of several minerals in milks. *Journal of Agricultural and Food Chemistry*, 47, 1520–1524.
- Rodriguez, E. M. R., Alaejos, M. S., & Romero, C. D. (2000). Concentrations of iron, copper and zinc in human milk and powdered infant formula. *International Journal of Food Sciences and Nutrition*, 51, 373–380.
- Soylak, M., Colak, H., & Turkoglu, O. (2006a). Heavy metal content of some cereals, spices and pulses from middle Anatolia region of Turkey. *Fresenius Environmental Bulletin*, 15, 345–348.
- Soylak, M., Colak, H., Turkoglu, O., & Dogan, M. (2006b). Trace metal contents of snack and appetizers consumed in Turkey. *Bulletin of Environmental Contamination and Toxicology*, 76, 436–441.
- Tripathi, R. M., Raghunath, R., Sastry, V. N., & Krishnamoorthy, T. M. (1999). Daily intake of heavy metals by infants through milk and milk products. *The Science of the Total Environment*, 227, 229–235.
- Vinas, P., Martnez, M. P., & Cordoba, M. H. (2000). Rapid determination of selenium, lead and cadmium in baby food samples using electrothermal atomic absorption spectrometry and slurry atomization. *Analytica Chimica Acta*, 412, 121–130.
- World Health Organization. (1982). Evaluation of certain food additives and contaminants. Twenty-sixth report of the joint FAO/WHO expert committee on food additives. WHO Technical Report Series No. 683. World Health Organization, Geneva.
- Yaman, M., & Cokol, N. (2004). Determination of trace elements in human milk, cow's milk, and baby foods by flame AAS using wet ashing and microwave oven sample digestion procedures. *Atomic Spectroscopy*, 25, 194–199.
- Yebra, M. C., Moreno-Cid, A., Cespón, R., & Cancela, S. (2004). Preparation of a soluble solid sample by a continuous ultrasound assisted dissolution system for the flow-injection atomic absorption spectrometric determination of iron in milk powder and infant formula. *Talanta*, 62, 403–406.