DETERMINATION OF SOME ESSENTIAL AND TOXIC ELEMENTS OF COMMERCIAL CHILDREN FOOD IN THE EGYPTIAN MARKETS

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ABSTRACT

The present work was carried out for determination of Antimony (Sb), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), Manganese (Mn), nickel (Ni), Tin (Sn) and zinc (Zn) in children food samples using inductively coupled plasma optical emission spectrometer (ICP-OES). The method showed that the quantification limits were varied between 0.02 and 1 mg/kg. The mean recoveries ± standard deviations at different spiking levels were varied between 81.95 ± 3.89 and 112.56 ± 3.31 % with coefficient of variation expressed as relative standard deviation ranged from 1.95 and 5.91%. The method trueness was confirmed by using three different certified references materials purchased from (WEPAL) and all obtained results were with in satisfactory ranges and had acceptable recovery and Z-score values. The method precision, in terms of relative standard deviation (RSD), was being below 5.16 % .The method uncertainty expressed as expanded uncertainty of all validated elements was found to be ≤ 22.68 %. The results obtained make the method suitable for accurate determination of validated elements in different kinds of children food samples at these low concentration values. Validated method was used for the determination of metallic contaminants in 50 samples covering 19 different brands of popular biscuits, snacks, cooked potato, bake, and cake in Egypt. The result showed that, all tested samples were free from any detectable amount of Pb. On the other hand, the resulting concentration ranges, in

mg/kg, were as follows: < 0.02–0.08 (Cd), < 1– 10.9 (Cu), 3.4 – 227.1 (Fe), 1.2 – 7.6 (Mn), 1.2 – 3.8 (Ni), 1 – 3.5 (Sn), and 3 – 269.1 (Zn).
Keywords: Children food; ICP-OES; Essential and toxic elements; Egypt.

INTRODUCTION

Many kinds of child-consumed candy products were sold at a low price at retail stores near elementary schools. Most of the packaging is so poorly designed that the inner coating does not maintain structural integrity, allowing ink components in the outer package layer to migrate into the candy. Furthermore, candy contact surfaces of the packages have a potential for contamination because finished packaging films are frequently distributed to end users in reel form in which the outer printed surface and food contact surfaces of the packages are in contact with each other. There are many kinds of metal-based inorganic pigments. The metal components can migrate from the printed surface to the food contact surface if the package is poorly designed. In particular, heavy metals from the ink can migrate to the candy if harmful lead (Pb) - or hexavalent chromium [Cr (VI)] – based inks are intentionally used in the food package. Migration can be severe if the surface of the candy is sticky. For more than a century, Cr (VI) exposure has been known to be associated with cancer induction in humans, especially bronchial carcinoma and lung cancer (Ki-Cheol *et al*., 2008; Khalil *et al*., 2016). However, not all the Cr (VI) compounds are equally potent as carcinogens. In particular, the water-insoluble Cr (VI) compounds of particulate forms are more potent carcinogens than the water-soluble ones (Patierno *et al*., 1998; Holmes *et al*., 2005).

Supplementation of metals in children food is currently a matter of discussion. Moreover, children food and foods may be hold toxic elements as a result of their natural presence in raw materials which be used, from contamination, or from food processing. For example, rice-based food products were recently reported to contain concentrations of arsenic above what is considered safe days (Meharg *et al*., 2008). Metals such as lead (Pb), cadmium (Cd), mercury (Hg), and arsenic (As) are considered toxic and are known to have deleterious effects, even in small quantities. Lead during pregnancy or early childhood will cause serious damage to the brain growth with the consequent loss of intellectual potential. Also, with prolonged intake of Cd may accumulate in the kidney and liver because of its long biological half-life and may lead to kidney damage (Ghuniem *et al*., 2019 a). Furthermore, the short term exposure to low level arsenic causes reduction in the production of leukocytes and erythrocytes, damage to blood vessels, nausea and vomiting, abnormal heartbeat, and pricking sensations in the hands and legs, its exposure for long time periods often leads to skin lesions, peripheral vascular disease, pulmonary disease and cardiovascular diseases, neurological problems, diabetes mellitus and certain types of cancers (Yoshida *et al*., 2004; Hopenhayn, 2006). Symptoms attributed to high level exposure to metallic mercury include lung damage, mucous membrane changes, vomiting, ausea, skin rashes, increased heart rate or blood pressure, renal dysfunction, and severe neurologic abnormalities (Asano *et al*., 2000; Ghuniem *et al*., 2019 b).

Therefore, the aim of this study is establishing and validating an analytical method for the determination of Pb, Cd, Sb, Cu, Zn, Fe, Cr, Sn, Co, Mn and Ni in a wide range of children food collected from Giza, Egypt.

MATERIALS AND METHODS

Apparatus: A Perkin Elmer Inductively coupled plasma optical emission spectrometer (ICP-OES) Optima 8300 coupled with ultrasonic nebulizer U5000 AT+ (CETAC). Milestone High-pressure microwave oven (Model: Ethos Up) purchased from Milestone – Italy. Water Purification System equipped with Q-POD Element coupled with Merck Millipore – Q® integral 5 (A10®) / Model : ZRXQ005T0 - USA. Mettler Toledo Top bench balance has range from 0.1 mg to 210 g.

Materials and Reagents: Certified reference metals stock standard solutions (1000 mg/L) of Pb, Cd, Sb, Cu, Zn, Fe, Cr, Sn, Co, Mn and Ni prepared in 2-3% HNO3 were purchased from Merck- Germany. Concentrated Nitric acid (HNO3) 65 % (w/w) was purchased from Merck - Germany. Emsure® Hydrogen peroxide (H2O2) 30 % was purchased from Merck - Germany. Water was deionised in the laboratory using Water Purification System equipped with Q-POD Element coupled with Merck Millipore – Q® integral 5 (A10®).

Sample collection: In this study, 50 children food samples corresponding to 19 different brands of biscuits, snacks, cooked potato, bake and cake were obtained randomly from local markets in Egypt. The samples consisted of 10 samples of each variety. The samples were coded and stored before analysis at conditions similar to those of retail shops. Constituents and samples types

of the children food samples are presented in Table 1.In this study, 3 different certified reference materials (CRM) (IPE 783 Wheat powder, IPE 998 Potato powder, and IPE 200 Maize powder) were purchased from Wageningen Evaluating Programmes For Analytical Laboratories (WEPAL) Netherlands.

Sample preparation: Homogenize the sample, weigh up to (0.5 g) of children food samples into the microwave digestion vessel; add 0.5 mL of deionized water to decrease the nitrous fumes resulting from the matrix digestion. Add 8 mL concentrated nitric acid to the digestion vessel and shake gently then add 2 mL of hydrogen peroxide. Adjust the microwave oven program as (Power = 1800 watt for 15 minutes until temperature reach 200 0C then let the temperature at 200 0C for 15 minutes, finally allow the microwave venting (Power = zero) until temperature < 80 0C). After the heating cycle have been completed, allow the vessels to cool down in a water bath for about 30 min then open the vessels carefully. Rinse down the lid and the walls with deionised water inside the vessel, then transfer the residual solution in 50 mL volumetric flask and completed it with deionized water to the marked volume.

Instrumentation: ICP-OES is started, with the water cooler before plasma igniting. The plasma must be ignited at least 30 minutes before wavelength calibration on optima spectrometers.. The measurement was carried out at optimize condition (plasma flow = 12 L/min, auxiliary flow = 0.2 L/min, nebulizer flow = 0.35 L/min, ICP radio frequency=1400 Watts, pump flow rate =2.5 L/min, view distance =15cm, ultrasonic nebulizer heater = 140 0C, ultrasonic nebulizer cooler = 3 0C and axial plasma view). ICP-OES was used for determination of Cu, Zn, Fe, Sn, Mn, Cr, Co, Cd, Pb, Sb, and Ni in

children food samples at different basic wavelengths which give higher sensitivity, lower relative standard deviation, sharpness peaks and no spectral interferences. The basic wavelengths were 205.56, 257.61, 238.204, 327.393, 202.548, 230.786, 227.02, 214.440, 220.353, 206.836 and 189.927 nm for Cr, Mn, Fe, Cu, Zn, Co, Ni, Cd, Pb, Sb, and Sn, respectively.

Analytical quality assurance: Analytical method validation of ICP-OES for analysis of Pb, Cd, Sb, Cu, Zn, Fe, Cr, Sn, Co, Mn and Ni in children food was assessed by determining several analytical parameters such as: limit of quantification (LOQ), method linearity, accuracy, and measurement uncertainty according to Eurachem (2014) and Eurachem / CITAC guidelines (2012).

Table(1): Constituents of children food samples

Item Name	Code	Constituents
Biscuits	A1	Wheat flour, sugar, Vegetable oil, fructose syrup, Sodium bicarbonate, and sodium acid
	A2	corn flour, milk, oil, and Glucose starch
	A3	Wheat flour, Sugar, Palm oil, fructose, salt, citric acid, ammonium& sodium, and bicarbonate de sodiumE503-E500
	A4	Wheat flour, Sugar, Palm oil, fructose, salt, citric acid, ammonium& sodium, and bicarbonate de sodiumE503-E500
	A5	wheat flour, sugar, Vegetable oil, Sodium bicarbonate, and sodium acid e500
	A6	wheat flour, veg oil, cocoa powder, salt, sugar, e500 why milk, palm oil, and vegetable oil
	A7	Wheat flour, sugar, refined, vegetable oils, skimmed milk powder, cocoa butter substitute, leavening agents, sodium bicarbonate and e500 vanilla
	A8	Wheat flour, date, sugar, Vegetable oil, palm, Glucose syrup, corn starch, sorbitol, bicarbonates and ammonium
	A9	Sugar, Vegetable oil, palm oil, wheat flour, cocoa powder, skimmed milk, whey milk, e450 vanilla
	A10	Wheat flour, Sugar, hydrogenated vegetable oil, cocoa powder , soya lecithin E322, potassium sorbateE211, salt, food flavour
Cooked potato	B1	Selected fresh potatoes, sun flower oil, salt, and sweet chilli seasoning
	B2	Selected fresh potatoes, sun flower oil, salt, and cheddar Shallats seasoning
	B3	Selected fresh potatoes, sun flower oil, and sea salt
	B4	Fresh potatoes, vegetable oil, chicken flavour, wheat derivative, and milk derivative
	B5	Fresh potatoes, refined palm olien oil, vinegar, and salt flavour
	B6	Fresh potatoes, refined palm olien oil, and spiced cheese flavour
	B7	Fresh potatoes, refined palm olien oil, tomato flavour
	B8	Fresh potatoes, refined palm olien oil, chilli lemon flavour
	B9	Fresh potatoes, vegetables oil, salt and vinegar, wheat and gluten
	B10	Fresh potatoes, vegetables oil, chilli lemon flavour, gluten
Snacks	C1	Corn, Vegetable oil, cheese flavour, gluten, lactose, and wheat
	C2	Corn, Vegetable oil, chilli flavour, gluten, lactose, and wheat
	C3	Corn, Vegetable oil, cheese flavour, gluten, lactose, and wheat
	C4	Corn gratis, Vegetable oil, chilli flavour, lactose, wheat, and gluten
	C5	Corn grits, Vegetable oil, Ketchup and hot dog flavour, Milk, Lactose, and wheat gluten
	C6	Corn, Vegetable oil, Corn flour, cheese flavour salt, and milk
	C7	Corn, Vegetable oil, Corn flour, cheese flavour salt, and milk
	C8	Vegetable oil, corn flour, salt, and peanut flavour
	C9	Vegetable oil, corn flour, salt, and cheese flavour
	C10	Vegetable oil, corn flour, salt, and cheese flavour

Cont. Table(1): Constituents of children food samples

Item Name	Code	Constituents
Bake	D1	Wheat flour, Water, palm olien, Cheese flavour, salt, sugar, yeast, and natural e160b
	D2	Wheat flour, Cheese flavour, Salt, water, and palm olien
	D3	Wheat flour, water, olien oil, yeast, and butter flavour
	D4	Wheat flour, water, olien oil, yeast, and cheese flavour
	D5	Wheat flour, water, olien oil, yeast, and salt flavour
	D6	Wheat flour, water, olien oil, yeast, and salt and vinegar flavour
	D7	Wheat flour, water, olien oil, yeast, and sweet chilli flavour
	D8	Wheat flour, water, olien oil, yeast, and pizza flavour
	D9	Wheat flour, ketchup flavour, Salt, water, palm olien, and yeast
	D10	Wheat flour, ketchup flavour, Salt, water, palm olien, and yeast
Cake	E1	Sugar, Wheat flour, hydrogenated Vegetable oil, egg, Coca , water and milk powder
	E2	wheat flour, sugar, whole, egg, milk powder, vegetable oil, cocoa, and water
	E3	Sugar, Wheat flour, hydrogenated Vegetable oil, egg, Coca, water and milk powder
	E4	Wheat flour, water, egg, vegtables oil, fructose, dextrose powder, whey powder and salt
	E5	Sugar, eggs, palm olien, hydrogenated, water, wheat flour, cocoa powder and wheat starch
	E6	Sugar, eggs, palm olien, hydrogenated, water, wheat flour, milk powder and wheat starch
	E7	Wheat Flour, Sugar, palm oil, Cocoa, Salt, glucose, and bicarbonate
	E8	Sugar, Vegetable oil, palm oil, wheat flour, egg, dextrose, cocoa and fructose syrup
	E9	Wheat flour, sugar, water, egg, cocoa, fructose, salt, Vegetable oil, and vanilla
	E10	Wheat flour, sugar, water, egg, cocoa, fructose, salt Vegetable oil, full cream and soya protein

RESULTS AND DISCUSSION

Method Validation: Validation was performed to ensure the reliability of the method. Before being used for quantitative analysis of Pb, Cd, Sb, Cu, Zn, Fe, Cr, Sn, Co, Mn and Ni in children food samples, the method was validated by determining some analytical parameters.

1) Practical limit of quantification (LOQ): The practical limits of quantification, expressed as the lowest validated spike level with acceptable criteria for trueness and precision. The minimum practical concentrations of tested elements in the analysed samples, which can be determined with acceptable accuracy were performed by analyse eight replicates at (1 mg/kg) for Mn, Cr, Co, Ni Fe, Cu, Sn and Zn, at (0.4 mg/kg) for Sb, at (0.05 mg/kg) for Pb, and at (0.02 mg/kg) for Cd. At these LOQs values the coefficients of variation expressed as relative standards deviations (RSD %) were found to be 4.75, 3.37, 4.10, 3.74, 4.00, 5.91, 4.58, 4.19, 3.91, 3.63, and 5.44 % for Cr , Co , Mn, Ni, Zn, Sn, Cu, Fe, Cd, Pb, and Sb, respectively.

2) Recovery test: The recovery tests were performed using 8 replicates of spiking soft drinks samples at 4 different concentrations levels. The spiking levels used for recovery test were at 1, 2, 5, and 10 mg/kg for Mn, Cr, Co, Ni Fe, Cu, Sn and Zn, at 0.4, 0.8, 1 and 2 mg/kg for Sb, at 0.05, 0.1, 0.5, and 1 mg/kg for Pb, and at 0.02, 0.1, 0.5, and 1 mg/kg for Cd. The mean recoveries ± standard deviations at different levels varied between 81.95 ± 3.89 and 112.56 ± 3.31 % with coefficient of variation expressed as relative standard deviation ranged from 1.95 and 5.91%.

3) Linearity: The values of correlation coefficients must be greater than 0.995 for accurate quantification as analytical response is linear over certain concentration ranges (Eurachem, 1998; Jeevanaraj *et al*., 2015).

(a) Linearity of the calibration curves: The dynamic linear range found to be linear from 0.05 up to 6 mg/L for Mn, Cr, Co, Ni Fe, Cu, Sn and Zn and

from 1 up to 100 µg/L for Pb, Cd, and Sb. All calibration curves correlation coefficients values were greater than 0.995 see Table 2.

(b) Linearity of the method: The linearity of the method was checked using 4 different spiking levels of soft drinks samples at 1, 2, 5, and 10 mg/kg for Mn, Cr, Co, Ni Fe, Cu, Sn and Zn, at 0.4, 0.8, 1 and 2 mg/kg for Sb, at 0.05, 0.1, 0.5, and 1 mg/kg for Pb, and at 0.02, 0.1, 0.5, and 1 mg/kg for Cd. The method linearity was found to be linear from LOQs values up to 10 mg/kg for Mn, Cr, Co, Ni Fe, Cu, Sn and Zn, from LOQs values up to1 mg/kg for Pb and Cd, and from LOQs values up to2 mg/kg for Sb. All the linearity of the method correlation coefficients values were greater than 0.995 see Table 2.

Table(2): Results of correlation coefficients of calibration curves and the linearity of the method.

Element	Dynamic linear range		Method linearity	
	Correlation coefficients	Slope	Correlation coefficients	Slope
Cr	0.99924	0.1232	0.9996	1.0621
Co	099954	0.1876	0.9999	1.0383
Mn	0.99988	0.4624	0.9998	0.9509
Ni	099963	0.0570	0.9999	0.9584
Zn	0.99940	0.2437	0.9995	0.8581
Sn	0.99966	0.04295	1.0000	1.0079
Cu	0.99957	0.1078	0.9982	0.8663
Fe	0.99988	0.5749	0.9984	0.8693
Cd	0.99982	0.5289	0.9986	0.8905
Pb	0.99989	0.07932	0.9993	0.9851
Sb	0.99990	0.1964	0.9998	0.9814

4. Method Accuracy: In this study, method accuracy was studied as two parameters trueness and precision (repeatability and reproducibility).

(a) Trueness: In this study, the trueness of the validated method was confirmed by using three certified reference materials (CRM) from WEPAL (IPE 783 Wheat powder, IPE 998 Potato powder, and IPE 200 Maize powder). Z-scores were calculated using the following equation:

$$Z - score = \frac{C_{Found} - C_{Assigned}}{S} \quad (1)$$

Z-score: a statistical measure that quantifies the distance a data point is from the mean.

C_{Found}: Found concentrations. S: Standard deviation from WEPAL.

$C_{assigned}$: Assigned values from WEPAL.

All measured results were within satisfactory range and had acceptable Z-score (-2≤ Z-score ≤ 2) see Table 3.

Table(3): Results of different certified reference materials from WEPAL.

CRM Type	Elements	Found Concentrations	Assigned values	Satisfactory Ranges			Units	Z-score
IPE 783 Wheat powder	Copper	3.48	3.31	2.38	-	4.24	mg/	0.37
	Iron	32.90	34.50	22.9	-	46.0	mg/	-0.28
	Manganese	17.94	19.00	15.2	-	22.8	mg/	-0.56
	Zinc	24.11	23.00	18.4	-	27.6	mg/	0.48
IPE 998 Potato powder	Copper	6.55	7.00	5.20	-	8.80	mg/	-0.50
	Iron	41.70	42.30	24.9	-	59.6	mg/	-0.07
	Manganese	5.51	5.42	3.49	-	7.35	mg/	0.09
	Zinc	19.02	19.30	14.9	-	23.6	mg/	-0.13
IPE 200 Maize powder	Cadmium	39.65	38.80	28.0	-	49.5	µg/	0.16
	Copper	2.74	2.59	1.74	-	3.44	mg/	0.35
	Iron	88.87	86.00	66.4	-	105.	mg/	0.29
	Lead	315.12	290.00	175.	-	405.	µg/	0.44
	Manganese	16.48	15.20	12.3	-	18.0	mg/	0.90
	Zinc	14.95	15.90	13.4	-	18.3	mg/	-0.78

(b) Precision: In this study, method of precision was confirmed by studding two component repeatability and reproducibility.

(I) Repeatability: The repeatability test was performed by analysing 8 replicates of fortified soft drink sample by the same operator and short intervals of time. The results of repeatability test expressed as relative standard deviation were found to be 3.99, 2.33, 2.67, 2.21, 3.03, 2.74, 2.26, 2.23, 3.96, 2.50, and 2.08 % for Cr, Co, Mn, Ni, Zn, Sn, Cu, Fe, Cd, Pb, and Sb, respectively.

(II) Reproducibility: In this study intra-laboratory reproducibility test was performed by analysing 30 replicates of fortified soft drinks samples (at 10 mg/kg for Cr, Co, Mn, Ni, Zn, Sn, Cu, and Fe, at 2 mg/kg for Sb, and at 1 mg/kg for Cd and Pb by different analysts on several days. The results of reproducibility test expressed as relative standard deviation were found to be 4.32, 3.21, 3.26, 3.25, 3.43, 2.73, 2.99, 3.47, 4.85, 4.94, and 5.16 % for Cr, Co, Mn, Ni, Zn, Sn, Cu, Fe, Cd, Pb, and Sb, respectively.

5. Measurement Uncertainty: Accumulated data from different quality control procedures was used for estimation of measurement uncertainty. Parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measured. The parameter may be, for example, a standard deviation or the width of a confidence interval. For estimating the overall uncertainty, it may be necessary to take each source of uncertainty and treat it separately to obtain the contribution of each source. Each of the separate contributions to uncertainty is referred to as an uncertainty component. When it expressed as relative standard deviation an uncertainty component

is known as relative standard uncertainty. The uncertainty component due to precision was investigated from intra-laboratory precision. The uncertainty component due to bias of the analytical procedure was investigated from recovery data using spiked samples, a significance test (t-test) was applied to test whether the recovery was significantly different from 100 % or not. The uncertainty component due to sample processing, which represent homogeneity of analyte in analytical sample according to codex guidelines was taken as a default value (10 %) (Codex Alimentarius Commission, 2003).The uncertainty due to reference standard preparation was estimated by accounting for reference standard purity, volumetric flask and pipettes using normal and triangle distributions. Normal distribution was used in case of calculation uncertainty due to reference stock standards purity, while triangle distribution was used in case of calculation uncertainty due to volumetric flask and pipettes. The uncertainty component due to reference standards preparations was found to be equal to 0.82 %. The total uncertainty combined standard uncertainty, equal to the positive square root of the sum of the squares of the individual uncertainty components as shown in the flowing equation (2):

$$U_{Combined} = \sqrt{(U_{precision})^2 + (U_{Bias})^2 + (U_{Reference})^2 + (U_{Sample\ Processing})^2} \quad (2)$$

Expanded uncertainty must be used for quantitative analysis in analytical chemistry. Expanded uncertainty was obtained by multiplying the combined uncertainty, by a coverage factor k, at confidence level of 95% (k = 2). The

measurement uncertainties expressed as expanded uncertainties were found to be 22.06 %, 21.14 %, 21.18 %, 21.18 %, 21.54 %, 20.83 %, 21.41%, 21.61 %, 22.56 %, 22.36 %, and 22.68 % for Cr, Co, Mn, Ni, Zn, Sn, Cu, Fe, Cd, Pb, and Sb, respectively see Table 4.

Table(4): Results of uncertainty components.

Element	Uncertainty Components					
	Standard Preparation	**Bias**	**Sample Processing**	**Precision**	**Combined Uncertainty**	**Expanded Uncertainty**
Cr	82.00%	1.51%	10%	4.32%	11.03%	22.06%
Co	82.00%	0.89%	10%	3.21%	10.57%	21.14%
Mn	82.00%	0.89%	10%	3.26%	10.59%	21.18%
Ni	82.00%	0.93%	10%	3.25%	10.59%	21.18%
Zn	82.00%	1.89%	10%	3.43%	10.77%	21.54%
Sn	82.00%	0.60%	10%	2.73%	10.42%	20.83%
Cu	82.00%	2.24%	10%	2.99%	10.71%	21.41%
Fe	82.00%	1.99%	10%	3.47%	10.80%	21.61%
Cd	82.00%	1.76%	10%	4.85%	11.28%	22.56%
Pb	82.00%	1.22%	10%	4.78%	11.18%	22.36%
Sb	82.00%	1.13%	10%	5.16%	11.34%	22.68%

Quality control

(a) Reagent blank: The reagent blank sample should be analysed with each set of sample. Reagent blank sample contain 8 mL of concentrated HNO_3 (65%) + 2 mL H_2O_2 (30%).

(b) Control samples: The performance of method is continuously tested using recovery test. Each analysed batch of soft drinks samples must contain control sample. Sample is spiked with suitable amount of standard solution to get 10 mg/kg for Cr, Co, Mn, Ni, Zn, Sn, Cu, and Fe, to get 2 mg/kg for Sb, and to get 1 mg/kg for Cd and Pb.

(c) Control charts: Control chart was used to monitor the stability of analytical precision. The results of control samples are recorded on the control chart. In case of individual point falls outside the stated limits

(upper control limit (UCL), upper warning limit (UWL) , mean central line , lower control limit (LCL) and lower warning limit (LWL)), the source or sources of error must be identified and corrected (Funk *et al.,* 1995).

Analyses of children food samples: Fifty children food having different brands were analysed by the validated method, and the results are presented in Table 5. Various concentrations of tested elements were detected in all samples. The results showed that the highest concentration of Cd, Cu, Zn, Fe, Mn, Sn, and Ni was determined in the sample numbers D9, D1, C7, D1, D1, C7, and C7, respectively.

Table(5): Elements compositions of the children food samples in (mg/kg).

Sample	Cr	Co	Cu	Fe	Mn	Ni	Zn	Sn	Cd	Pb	Sb
A1	ND	ND	2.5	14.6	4.4	< 1	5.9	< 1	ND	ND	< 0.4
A2	ND	ND	1.1	91.6	2.4	ND	4.3	< 1	ND	ND	< 0.4
A3	ND	ND	ND	6.5	3.2	ND	3.5	< 1	ND	ND	ND
A4	ND	ND	< 1	8.6	3.4	< 1	4.3	< 1	ND	ND	< 0.4
A5	< 1	ND	3.2	58.4	3.6	ND	33.5	< 1	ND	ND	< 0.4
A6	< 1	ND	2.4	12.3	5.4	< 1	8.9	< 1	ND	ND	< 0.4
A7	< 1	< 1	5.9	49.8	6.7	< 1	14.7	< 1	< 0.02	ND	< 0.4
A8	ND	ND	< 1	7.1	2.3	ND	3	< 1	ND	ND	ND
A9	ND	< 1	4.7	45.1	4.9	1.4	11	< 1	< 0.02	ND	< 0.4
A10	ND	ND	3.9	17.1	3.6	< 1	8.7	< 1	ND	ND	ND
B1	< 1	ND	4.9	10.4	4	ND	6.9	ND	< 0.02	ND	< 0.4
B2	ND	< 1	5.5	8.5	3.2	< 1	9.5	< 1	< 0.02	ND	< 0.4
B3	ND	ND	4.8	9	3.4	ND	9.3	< 1	0.02	ND	< 0.4
B4	< 1	ND	6.4	6	3.7	< 1	6	< 1	0.04	ND	< 0.4
Sample	Cr	Co	Cu	Fe	Mn	Ni	Zn	Sn	Cd	Pb	Sb
B5	ND	< 1	7.9	23.5	4	< 1	11	< 1	< 0.02	ND	< 0.4
B6	ND	< 1	8.3	11.2	3.7	< 1	9.7	< 1	< 0.02	ND	< 0.4
B7	ND	< 1	6.7	10.8	3.4	< 1	9	< 1	ND	ND	< 0.4
B8	ND	ND	4.6	14.8	4.2	ND	9	< 1	ND	ND	< 0.4
B9	ND	< 1	3.6	8.5	3.01	ND	4.6	< 1	< 0.02	ND	< 0.4
B10	< 1	ND	3	10.9	2.6	< 1	6.4	ND	< 0.02	ND	< 0.4
C1	< 1	ND	ND	17.9	1.2	1.6	33.4	< 1	ND	ND	ND
C2	ND	ND	1.7	22.2	1.9	< 1	31.9	1.2	ND	ND	< 0.4
C3	ND	ND	ND	20.74	1.7	< 1	39.7	1	ND	ND	< 0.4
C4	ND	ND	4.6	16.7	2.3	ND	58.2	1.8	ND	ND	< 0.4

Cont. Table(5): Elements compositions of the children food samples in (mg/kg).

Sample	Cr	Co	Cu	Fe	Mn	Ni	Zn	Sn	Cd	Pb	Sb
C5	ND	ND	ND	13.2	1.2	< 1	55.9	< 1	ND	ND	ND
C6	ND	ND	ND	24.7	1.2	2.5	50.3	< 1	ND	ND	ND
C7	ND	ND	9.9	26.1	2.1	3.8	269.1	3.5	ND	ND	< 0.4
C8	< 1	ND	< 1	3.4	3.2	< 1	76.7	1.1	ND	ND	< 0.4
C9	< 1	ND	< 1	16.5	1.8	< 1	67	< 1	ND	ND	< 0.4
C10	ND	ND	ND	20	1.8	< 1	53.5	< 1	ND	ND	< 0.4
D1	< 1	ND	10.9	227.1	7.6	ND	12.1	2	< 0.02	ND	< 0.4
D2	ND	ND	< 1	20.3	2.6	ND	5.1	< 1	< 0.02	ND	ND
D3	ND	ND	10.6	52.3	6.2	< 1	12.7	2	< 0.02	ND	< 0.4
D4	ND	ND	2.5	16.3	3.9	< 1	6.1	< 1	ND	ND	ND
D5	ND	ND	1.7	32.3	4.5	< 1	6.6	< 1	ND	ND	ND
D6	ND	ND	ND	36.7	4.1	< 1	5.8	< 1	ND	ND	ND
D7	ND	ND	1.3	55.4	5	ND	6.7	< 1	< 0.02	ND	< 0.4
D8	ND	ND	< 1	40.9	4.3	ND	7.2	< 1	0.05	ND	< 0.4
D9	ND	ND	< 1	31.1	5.7	< 1	10.7	< 1	0.08	ND	< 0.4
D10	ND	ND	ND	25.5	2.7	< 1	5.8	< 1	ND	ND	ND
E1	ND	ND	1.6	12	2.2	< 1	7	< 1	ND	ND	ND
E2	ND	ND	2.3	15	2.6	< 1	6.6	< 1	ND	ND	< 0.4
E3	ND	ND	2.2	48.9	4	1.2	7.2	< 1	ND	ND	< 0.4
E4	< 1	ND	2.4	36.2	3.1	< 1	7.9	< 1	ND	ND	ND
E5	ND	< 1	5	52.4	6.1	< 1	14.7	< 1	ND	ND	< 0.4
E6	ND	ND	3.3	7.8	2.5	< 1	6.3	< 1	< 0.02	ND	< 0.4
E7	ND	ND	ND	17.5	1.6	< 1	31	< 1	ND	ND	< 0.4
E8	ND	ND	1.9	5	2.4	ND	8.9	< 1	ND	ND	< 0.4
E9	< 1	ND	3.9	15.9	3.1	ND	10.1	< 1	ND	ND	< 0.4
E10	< 1	ND	6	23.1	1.3	ND	4.4	1.3	ND	ND	< 0.4
Maximum Limits (mg/kg)	-	-	-	-	-	-	-	50	0.04	0.05	-

Antimony is a cumulative toxic element with obscure biological function and its physicochemical and toxic properties depend on its binding form and oxidation state. Antimony trioxide (Sb2O3) is used as a catalyst in the manufacture of polyethylene terephthalate (PET) worldwide. Sb2O3 is a suspected carcinogen and is listed as a priority pollutant by EU and the USEPA (Ross and Adrian, 2009; Ghuniem *et al*., 2019 c). In this study, 74% of the total numbers of analysed samples have detectable amount of

antimony, but all these concentrations levels were less than quantification limit in various brands of children food samples.

Cadmium is the seventh most toxic heavy metal as per Agency for Toxic Substances and Disease Registry (ATSDR) ranking. Cadmium is also present as an impurity in several products including fertilizers, pesticides, detergents and refined petroleum products. Some possible food sources of cadmium are in peanuts, soybeans, rice, medicinal herbs, lettuce, corn, oats, wheat, spinach, fish, shrimps and mushroom. Exposure to cadmium at very high levels can result in serious health problems related to bone defects in humans and animals liver and kidneys and can cause death (Monisha *et al*., 2014; Raja and Namburu, 2014). The concentration of cadmium in this study was ranged between (< 0.02 and 0.08 mg /kg) in the various brands of children food . The highest concentration of cadmium was found in bakes samples. On the other hand, all snacks samples were free from any detectable amount of Cd.

Chromium is used in a number of industrial applications, including industrial water cooling, electroplating, tanning, paper pulp production, and petroleum refining. The Cr (III) is biologically essential for its role in protein and sugar metabolism, while Cr (VI) is potentially toxic and carcinogenic, and has adverse impact on metabolic processes (Mandina and Tawanda, 2013; Swapnil *et al*., 2017). In this study, 26 % of the total numbers of analysed samples have detectable amount of chromium, but all these concentrations levels were less than quantification limit in various brands of children food samples.

Cobalt and its compounds are widely distributed in nature and are part of numerous anthropogenic activities. Cobalt is an essential metal that is found in the active site of B12 vitamin and represents an important role in biochemical reactions of life. Cobalt sources were allocated to four exposure settings: dietary, environmental, occupational, and medical exposure. Oral intake of cobalt supplements and internal exposure are the main source of the highest systemic cobalt concentrations inside human body. Excessive exposure has been shown to induce various adverse health effects. Toxicological effects of cobalt include vasodilation, flushing and cardiomyopathy in animals and human (Claudia *et al*., 2017; Laura *et al*., 2017). In this study, 16 % of the total numbers of analysed samples have detectable amount of cobalt, but all these concentrations levels were less than quantification limit in various brands of children food samples.

Copper is one of the essential heavy metals found in all animals and food chain and environment, including soil and water and it is an essential nutrient for humans and animals in small amounts. The biological functions of copper include red blood cell synthesis, normal iron metabolism, cell metabolism; connective tissue metabolism and bone development. Acute poisoning from intake of excessive copper can cause vomiting, temporary gastrointestinal distress with symptoms such as nausea and abdominal pain. Exposure to high doses of copper can cause liver toxicity that resulted in death (Sevcikova *et al.*, 2011; Manju, 2015; Izah *et al*., 2016). The concentration of copper in this study was ranged between (< 1 and 10.9 mg /kg) in the various brands of children food . The highest concentration of copper was found in bake samples. 30 % of bake samples have copper contents below the quantification

limit, while 20 % of biscuit and snacks samples have copper contents below the quantification limit. On the other hand, all analysed cooked potato samples have copper concentrations levels above the quantification limit with mean concentrations (5.57 mg/kg), while 90 % of analysed cake samples have copper concentrations levels above the quantification limit with mean concentrations (2.86 mg/kg).

Iron is the fourth most abundant element which constitutes the most of the part of earth crust. Iron is essential for almost every organism due to its involvement in a wide variety of metabolic processes. The most common disease resulting from iron deficiency is anaemia in human. While excess intake of iron can cause several health implications such as an increased risk for heart diseases, cancer and endocrine problems, arthritis, diabetes and liver disease (Ramesh et al., 2016; Chitra *et al*., 2017). The concentration of iron in this study was ranged between (3.4 and 227.1 mg /kg) in the various brands of children food. The highest concentration of iron was found in snacks samples, while, the lowest concentration of iron was found in bake samples. The mean concentrations of iron in biscuit, cooked potato , snacks, bake, and cake were found to be 31.11, 11.36, 18.14, 53.79, and 23.38 mg/kg, respectively.

Lead is a highly toxic metal whose widespread use has caused extensive environmental contamination and health problems in many parts of the world. Lead breaks the blood-brain barrier and interferes with the normal development of brain in infants (Andre *et al*., 2005; Raja and Namburu, 2014). In this study, all analysed samples of the various brands of infant formulae were free from any detectable amount of lead (Pb).

Manganese is the twelfth most abundant element in the earth crust and is naturally present in food, water, soil and. rocks. Manganese is an essential nutrient present in several food items for humans, plants and animals and is required for development, growth and maintenance of health. Ingestion of manganese from drinking water may pose significant risks for children's health, especially neurodevelopment (Santamaria, 2008; Valfredo *et al*., 2009; Maryse *et al*., 2018). The concentration of manganese in this study was ranged between (1.2 and 7.6 mg /kg) in the various brands of children food . The highest concentration of manganese was found bake samples, while the lowest concentration of manganese was found snacks samples. The mean concentrations of iron in biscuit, cooked potato, snacks, bake, and cake were found to be 3.99, 3.52, 1.84, 4.66, and 2.89 mg/kg, respectively.

Nickel is an essential micronutrient for microorganisms, plants and certain mammals which present in a wide range of primary crops, animals and foodstuffs. Nickel is used in many industrial applications such as the manufacturing of stainless steel. Occupational exposure has been shown to give rise to elevated levels of nickel in urine, blood and body tissues (Aleksandra and Urszula, 2008; Francesco *et al*., 2009; Pizzutelli, 2011). In this study, 58 % of the total numbers of analysed samples have detectable amount of nickel less than quantification limit in various brands of children food samples, while 10 % of analysed samples have nickel concentrations levels above the quantification limit with mean concentrations (2.1 mg/kg).

Tin is one of the toxic metals, which could be accumulating in the human body and animals tissues. Tin may be released into the atmosphere from windstorms, volcanic emission, forest fires, roads, and farming activities.

Exposure to a large amount of tin in canned food is taken daily over a long period, acute effects such as stomach aches, anaemia, occur problems in liver and kidney (Cima, 2011; Hamid and Homeira, 2013). In this study, 80 % of the total numbers of analysed samples have detectable amount of tin less than quantification limit in various brands of children food samples, while 16 % of analysed samples have tin concentrations levels above the quantification limit with mean concentrations (1.74 mg/kg).

Zinc is one of the essential elements for living cells for many organisms and zinc deficiency has been associated with multiple adverse conditions, including impaired immune function. Excess amount of zinc can pose serious toxicity to human health as well as to the ecosystem, therefore, zinc is consider as both an essential and potentially toxic metal. Zinc is widely distributed in different food such as meats, fish, poultry, cereals and dairy foods. Oral zinc supplementation can reduce the effects of the common cold; however, there is strong clinical evidence that intranasal zinc gluconate gel treatment for this purpose causes anosmia or the loss of the sense of smell among people who had used intranasal zinc sprays and gels (Heidi *et al*., 2016; Man., 2017). The concentration of zinc in this study was ranged between (3 and 269.1 mg /kg) in the various brands of children food . The highest concentration of zinc was found in biscuit samples, while, the lowest concentration of iron was found in snacks samples. The mean concentrations of zinc in biscuit, cooked potato, snacks, bake, and cake were found to be 9.78, 8.14, 73.57, 7.88, and 10.41mg/kg, respectively.

CONCLUSION

In this study, ICP OES technique was used to validate the analytical method for the determination of trace and toxic element s in children food. The validated methodology proved to be fast, easy and simple, and so it can be very useful for routine laboratories. The method quantifications limits were found to be much lower than the maximum permissible limits of metal contaminants stated by Egyptian, WHO and European standards in food. This method can be suitable and applied for all kinds of children food. This method can be recommended for the Egyptian standard organisation for the determination of Pb, Cd, Sb, Cu, Zn, Fe, Cr, Sn, Co, Mn and Ni i in children food as well as help with the elucidation of toxicological studies, which may be interesting for health. A study of the levels of 11 trace and toxic metals was conducted using a total of 50 samples of children food collected from supermarkets Giza, Egypt. The analysis of the data indicated that all samples were free from any detectable amount of Pb while, different concentrations levels of Cd, Cu, Zn, Fe, Sn, Mn and Ni were quantified in the analysed children food sample. The data from this study will be useful to health authorities as well as researchers interested in epidemiological studies.

RECOMMENDATIONS

Microwave digestion method was especially recommended for fast, easy and simple, and so it can be very useful for routine laboratories for determination of lead (Pb), cadmium (Cd), antimony (Sb), copper (Cu), iron (Fe), zinc (Zn), chromium (Cr), manganese (Mn), tin (Sn), cobalt (Co) and nickel (Ni) in children food samples. Also hyphenation between ultrasonic

nebuliser with inductively coupled plasma optical emission spectrometer can make the method suitable for accurate determination of elements in different kinds of children food samples even at low concentration values.

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تقدير بعض العناصر الضرورية والسامة في أغذية الأطفال المتاحه في الأسوق المصرية

[٥]

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المستخلص

في هذه الدراسة، تم التحقق من صحة طريقة تحليل سريعة وسهلة وبسيطة لتحديد العناصر الاتية الأنتيمون (Sb) والكادميوم (Cd) والكروم (Cr) والكوبالت (Co) والنحاس (Cu) والحديد (Fe) والرصاص (Pb) المنجنيز (Mn) والنيكل (Ni) والقصدير (Sn) والزنك (Zn) في عينات أغذية الأطفال باستخدام جهازالحث بالبلازما المقترن بمطياف الانبعاث البصري (ICP-OES). تمت دراسة حدود الكميات العملية، الخطية (النطاقات الديناميكية الخطية وطريقة الخطية)، الدقة (الصدق والكفأه)، ومعاملات عدم اليقين في القياس. أظهرت الطريقة أن حدود القياس الكمي كانت تتراوح بين ٠,٠٢ و ١ مللي جرام / كيلوجرام. وقد تراوحت متوسطات معدلات استرجاع النتائج عند انحرافات المعيارية المختلفة ما بين ٨١,٩٥ ± ٣,٨٩ و ١١٢,٥٦ ± ٣,٣١ ٪ مع معامل الاختلاف المعبر عنه بالانحرافات المعيارية النسبية التي تراوحت ما بين ١,٩٥ و ٥,٩١ ٪. تم التأكيد علي صحة الطريقة من خلال استخدام ثلاث مواد مرجعية مصدقة مختلفة تم شراؤها من (WEPAL) وكانت جميع النتائج التي تم الحصول عليها في نطاقات مرضية وكان لها قيم مقبولة للاسترجاع. وقد كانت دقة الطريقة، من حيث الانحراف المعياري النسبي (RSD)، أقل من ٥,١٦ ٪. وقد تم العثور على عدم اليقين في طريقة المعبر عنها بعدم اليقين الموسع لجميع العناصر التي تم التحقق من صحتها في الطريقة ≥ ٢٢,٦٨ ٪. النتائج التي تم الحصول عليها تجعل الطريقة مناسبة لتحديد دقيق للعناصر تم التحقق من صحتها في أنواع مختلفة من عينات أغذية الأطفال حتي لو كانت بتركيزات منخفضة. وقد تم استخدام الطريقة التي تم التحقق من صحتها في تحديد الملوثات المعدنية في ٥٠ عينة تغطي ١٩ علامة تجارية مختلفة في مصر من البسكويت والمقرمشات والشبسي والمخبوزات والكيك. أظهرت النتيجة أن جميع العينات التي تم اختبارها كانت خالية من أي كمية يمكن اكتشافها من الرصاص. من ناحية أخرى، كانت نطاقات التركيزات الناتجة، بالمللي جرام / كيلوجرام، على النحو التالي: < ٠,٠٢– ٠,٠٨ (الكادميوم)، < ١– ١٠,٩ (النحاس)، ٣,٤ – ٢٢٧,١ (الحديد)، ١,٢ – ٧,٦ (المنجنيز)، ١,٢ – ٣,٨ (نيكل)، ١ – ٣,٥ (قصدير)، و٣ – ٢٦٩,١ (زنك).

الكلمات الدالة: طعام الأطفال، جهازالحث بالبلازما المقترن بمطياف الانبعاث البصري؛ العناصر الضرورية والسامة، مصر.