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Major and trace elemental analysis in milk powder by inductively coupled plasma-optical emission spectrometry (ICP-OES) and instrumental neutron activation analysis (INAA)

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Abstract: Major and trace element in seven different kinds of milk powder were studied. The concentration of 24 elements were determined by ICP-OES method, from these elements 9 elements determined by INAA. The determination of trace element contents of foodstuffs, especially milk as daily drink for all peoples age which being a complex food has great importance. The elemental analysis of milk is important both as an indicator of environmental contamination and because milk is a significant pathway for toxic metal intake and a source of essential nutrients for humans. The major elements are Ca, K, Mg, Na, P and S. While trace element are B, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Se, Sn., Sr., V., W and Zn.,

ICP-OES technique is shown to be a powerful tool for trace determinations in powder samples. This is shown by its use for analysis of a series of the milk powders mentioned and comparative results of other direct technique such as instrumental neutron activation analysis.

Analysis of both standard reference material A-11 milk powder and NBS Orchard leaves for quality accurance bad been completed, and used for a relative method calculate. The importance of the major and trace elements to human health was discussed.

Keywords; milk powder; ICP-OES; INAA

Introduction

The determination of trace element contents of food stuffs especially of milk which being a complex food has great importance. The milk is daily intake for all kind of pepole small and bige. The quality of foods is determined by the proportion of the individual components such as protein, fat, vitamins, hydrocarbons and by the major sources of Ca and P.

The importance of trace elements in nutrition is now widely recognized as the list of elements are indispensable to the proper nutrition is increasing steadily. In addition to many of the essential elements to human health presented in agricultural products, some toxic elements for the human biosystem may also be taken up from the environmental pollution.

Milk is basic food for both big and adults (kides). At least, every day, every body assumed to drink a cup of milk with medium sige. This basic milk taken from different animals which live on different plants. During growing these plants may be pollued by toxic elements and heavy metals from fertilyzers (El-Ghawi, 1999).

So a definite progress has been achieved in the study of trace elements composition of milk following the improvement in the analytical techniques that could yield reliable results at low concentration levels.

The determination of minor and trace elements in the milk powder is very important due to its high consumption by Egyptian population and its indicator of environmental contamination, a signicant pathway for toxic metal intake by humans and a source of essential nutrients. Previously, milk liquid and milk powder analysis was carried out using flame and graphite furnace atomic absorption spectrometry and even anodic stripping voltammetry (Kluckner, 1981). Inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS) are now preferred for routine determinations because of the repid multi-element analysis capabilities of these techniques (Munter, 1979; Barnett, 1983; Borkowska, 1996; Emmett, 1988; Dean, 1987). The ICP-MS technique has the advantage of sensitivity to measure toxic trace elements but is unable to analyze high dissolved solid contents for long perids of time and the instrumentation is more expensive than ICP-OES.

Nuclear analytical techniques, with their broad band of applicability to almost all matrix types and their exceptional sensitivity to many elements are an indispensable tool for environmental research. Neutron activation analysis and also inductively coupled plasma(ICP-OES) optical emission spectrometry, appears to be an attractive techniques for determining major and minor elements in the milk powder(Ryan, 1997; Andrew, 1999; Cunha, 1996; Bejey, 1995; Al-Jobori, 1990; Maihara, 1988).

In the present work, different kind of milk powder samples are collected from local marked in Egypt, analyzed by the most sensitive method of NAA and also by ICP-OES for determin the major, minor, trace and toxic elements.

This work describes the direct analysis of milk powder using standard quantitative calibration with aqueous standards. The accuracy and validity of the method was assessed by the use of National Institute of Standards (NIS) Standard Reference Material (SRM) A-11 milk powder.

1 Experimental

1.1 Sample preparation for neutron activation analysis

Seven samples of milk powder from local marked in Egypt were prepared for neutron activation analysis. 0.1 g from each sample was sealed in clean aluminum foil that was used as a container. Three replicates were taken from each sample. An empty Al foil of known weight is irradiated as well under the same conditions for calculating the background. Also the standard reference material (USDC, 1971) NBS 1571 orchard leaves and A-11 were irradiated at the same condition, for a relative calculation and for analytical quality assurance testing.

For measuring the thermal flux in the site of irradiation, 0.03 g gold sheet was wrapped in the same type of Al-foil. All samples and the standard reference material were irradiated for 4h at an average thermal neutron flux of $5.6 \times 10^{13} \text{ n/(cm}^2 \cdot \text{s})$ in the Second Research Egyptian Reactor(ET-RR-2). The samples were left to cool for about one week. A gamma ray spectrum of each irradiated sample was collected for 2h and repeated weekly for one month. The data were then corrected for background.

γ-ray spectra were collected by high resolution hyper pure germanium detector which have a resolution 1.9 keV at the 1332.5 keV photo-peak of ⁶⁰ Co. The peak to compton ratio was 55-6 and the detection efficiency was 30%. The energy and efficiency calibration curves up to about 3 MeV and the experimental conditions were carried out using the multi-gamma ray standard sources MCS-4 (Nuclear Measurement Group, 1998).

For accuracy, the same samples were sent to the Plant Health and Soil Conservation Station of Budapest, Department of Environmental Protection, Hungary for a comparison, using inductively coupled plasma optical emission spectrometry (ICP-OES).

1.2 Preparation of samples for ICP-OES analysis

For the ICP-OES analysis the samples have to be presented in the form of an aqueous solution. Ideally, complete dissolution is a prerequisite for accurate analytical results. The milk powder samples have been digested by a mixture of nitric acid and hydrogen peroxide in an insert of PTFE digestion bomb at 130 °C for 4 h. Two parallel samples of 700 mg milk powder were measured into a glass flask on an analytical balance, then 4 ml of high purity nitric acid (HNO3 65%), and 1.5 ml of hydrogen peroxide (H2O2 30%) was added to the sample. The flask was placed into a block heater and heated below the boiling point of the acid to 120°C. After about five minutes heating of the samples evolution of nitrous gases was observed indicating the start of the digestion reaction. At this time the inserts were removed from the heater and left for about ten minutes to react. When the fast reactions of the digestion have been slowed down the inserts were put back to the heater for an other run. After this predigestion stage the inserts were put into the pressure bomb and the main section of the digestion was done at 130 °C for 4 h under pressure in closed vessel. After this the bombs were cooled down and opened.

Table 1 Operating conditions of ICP-OES measurement

Instrument	Plasma labtest ICP spectromete
Plasma (Ar-Ar)	27.12 MHz
Power	1.3 kW
Outer argon	12 1/min
Intermediate argon	1.2 1/min
Inner argon	0.8 1/min
Observation height	13 mm
Sample flow rate	3 ml/min
Integration time	5 s
Sample introduction	By Gilson minipuls pump

The sample solution was quantitatively transferred into ²⁷ 25 ml volumetric flask and filled up with deionised water. Later the solutions were filtered into dry polyethlene flasks.

The ICP-OES measurements were carried out by a plasmalab ICP-OES instrument under the Labtes specification of the operating conditions which given in Table 1. The instrument consist of 40 channel Pashen-Runge type vacuum polychromator with PM-tube detectors. The argon-argon plasma source is working at 27.12 MHz

with maximum power of 2.0 kW. In our measurements it was adjusted to 1.3 kW. The sample solution was pumped by Gilson Minipuls peristaltic pump at a flow rate 2.8 ml/min.

Results and discussion

To confirm the accuracy of the detection system, the results of some elements which are well resolved in the reference material A-11 milk powder are compared with the certified values and the calculation of the Table 2, show good agreement with the certified values, and recoveries of more than 90% have been achieved.

The concentration of the elements for different types of milk powder represented in Tables 3-6 for big people and Tables 7-9 for milk powder of childeren. 24 elements were determined by ICP-OES

recoveries percentage. The results given in Table 2 Comparison between the present work and the certified values for the standard material milk powder A-11 (ppm), also the percentage recoveries

Elements	Certified Y	Present work X	Recoveries ($X/Y \times 100\%$)
Ca	12900.0	12100.0	93.790
Cr	0.018	0.016	88.890
Fe	3.650	3.550	97.260
Mo	0.092	0.110	119.570
Rb	30.800	31.200	101.290
Se	0.034	0.032	94.120
Zn	38.900	39.100	100.510

while 9 element were determined by INAA for the comparison between the two techniques, these elements are long half-life isotopes which are measured by the delayed technique. The concentration of the major elements Ca, K, Mg, Na, P and S were determined by the absolute method (Eissa, 1998) as well as by the relative method (Erdtmann, 1986).

The concentration of trace elements (Al, B, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Se, Sn, Sr, V, W and Z) were determined by ICP-OES and some of them, also by INAA. The results are given for all analytical techniques in $\mu g/g$ concentration units and uncertainties of counting are calculated in percent (Tables 3-6).

Table 3 Element concentrations of Nido milk powder

Table 4 Element concentrations of Karnchian milk powder

	P-OES	C) The									
		SD	RSD	INAA	SD	Element	ICP-OES	SD	RSD	INAA	SD
Al <	3.85	1.88	48.83	-	-	Al	< 2.25	1.19	53.02	-	-
В 2	2.40	0.61	25.41	-	-	В	4.00	1.50	37.50	-	-
Ba (0.83	0.38	45.78	-	-	Ba	0.60	0.11	18.33	-	-
Ca 98	845.0	71.0	0.72	9920.0	297.60	Ca	9500.0	130.0	1.37	9621.0	288.63
Co <	0.25	0.10	40.00	0.15	0.01	Co	< 0.23	0.05	22.22	0.35	0.02
Cr (0.28	0.15	50.0	0.45	0.02	Cr	0.20	0	0.0	0.23	0.01
Cu (0.70	0.5	71.43	-	-	Cu	0.80	0.19	23.75	-	•
Fe 4	4.30	2.30	53.49	5.80	0.29	Fe	3.80	0.83	21.84	4.30	0.22
K 18	3450.0	208.0	1.12	-		K	18100.0	280.0	1.55	-	-
Li	< 4	0	0	-	_	Li	< 4	0	0	-	_
Mg 8	392.0	8.80	0.99	-	-	Mg	840.0	14.00	1.67	-	-
Mn (0.51	0.55	107.84	-	-	Mn	0.30	0.19	63.33	_	_
Mo (0.50	0.18	36.40	0.70	0.04	Mo	0.35	0.10	28.57	0.41	0.02
Na 30	082.5	28.0	0.91	-	_	Na	2830.0	44.0	1.55	-	-
Ni <	0.50	0.15	30.0	-	_	Ni	0.40	0	0.0	_	-
P 14	442.5	17.0	1.18	-	-	P	1430.0	14.0	0.98	-	-
S 2:	595.0	31.0	1.19	-	-	\mathbf{s}	2620.0	22.0	0.84	-	-
Sb	< 2	0	0	1.20	0.06	Sb	2.0	0	0	3.20	0.16
Se	< 3	0	0	4.10	0.20	Se	< 3	0	0	4.50	0.23
Sn 4	4.70	0.50	10.64	-	-	Sn	4.20	0.17	4.15	-	
Sr (0.01	0	0	-	-	Sr	0.01	0	0	~	
V <	0.20	0	0	-	-	\mathbf{v}	< 0.37	0.12	32.43	-	-
W <	4.25	0.95	22.35	2.40	0.12	W	< 2.75	0.95	34.55	3.10	0.16
Zn	30.8	0.51	1.66	32.20	1.61	Zn	30.50	0.30	0.98	31.90	1.59

Notes: the unit in Table 3-Table 4 is ppm

2.1 Calcium(Ca)

The concentration of calcium(Ca) in the samples have been varied from 10200 ppm in Gratyzia milk powder to 4050 ppm in Nstogeen milk powder. The highest concentration found in the milk for big people while the concentration of Ca in the milk for childeren is lees to the half. Ca is major element in all types of milk, and its important for the human health. It is a major mineral of bone and teeth. It is invalued in blood clotting and improvement of body immune defences.

2.2 Potassium(K) and sodium(Na)

The concentration of potassium in the milk samples ranging from 18550 ppm in Gratyzia milk powder to 8390 ppm in cemalak milk powder. While sodium(Na) concentration ranged between 3080 ppm in Nido milk to 1200 ppm in Nstogeen milk powder. Sodium and potassium maintain normal fluid blance inside and outside the cells. Potassium if taken simultaneously with Na prevents increase the blood pressure.

2.3 Phosphorus(P)

The concentration of (P) ranged from 1475 ppm in Gratyzia milk powder to 469 ppm in Nstogeen. Phosphorus is very important for the human health, it forms with calcium the formation of bone and teeth, also it is found in all body cells as well as in blood and muscles.

2.4 Silphur(S)

The concentration of (S) ranged from 2620 ppm in Karnchian milk powder to 1260 ppm in cemalak milk powder. Silphur has important function in protein structure and also in enzymatic activity.

The concentration of B ranged from 4—1 ppm, Ba ranged from 0.91—0.11 ppm, Cr concentration ranged between 0.3—0.2 ppm and Cu ranged from 3.8 to 0.3 ppm.

From the results its clear that copper(Cu), in milk powder of children is higher than the milk powder of big people. The concentration of Fe ranged between 68.6—3.3 ppm, also Fe in milk for children is high. Copper is a factor necessary for the absorption and use of Iron(Fe) in the formation of hemoglobin and also used in many enzymes.

Mn concentration ranged between 0.51—0.08 ppm and Mo ranged between 0.5—0.3 ppm. The concentration of Sn ranged between 7—4 ppm, while the concentration of Sr and Zn ranged from 0.54—0.01 ppm and 41.2—29.9 ppm respectively.

The concentration of trace elements Al, Co, Li, Ni, Sb, Se, V and W are less than the detection limit of ICP-OES method.

Table 5 Element concentrations of Freyzyana milk powder

Table 6 Element concentrations of Gratyzia milk powder

Element	ICP-OES	SD	RSD	INAA	SD	F1	ICP-OES	SD	RSD	INAA	SD
				INAA	317	Element				INAA	317
Al	< 1	0	0	_	-	Al	2.55	1.06	41.57	-	-
В	1.20	0.49	40.83	***	-	В	1.10	0.14	12.73	-	-
Ba	0.28	0.02	6.07	-	-	Ba	0.91	0.01	0.78	-	-
Ca	9670.0	62.0	0.64	9710.0	291.30	Ca	10200.0	0	0	11321.0	339.60
Co	< 0.20	0	0	0.38	0.02	Co	0.20	-	_	0.33	0.02
Ст	< 0.20	0	0	0.35	0.02	Cr	0.20	-	-	0.28	10.0
Cu	0.60	0.14	23.33	-	_	Cu	< 0.3	-	-	-	-
Fe	5.00	1.50	30.0	7.80	0.39	Fe	3.35	0.21	6.27	4.20	0.21
K	17800.0	130.0	0.73	-	-	K	18550.0	70.0	0.38		-
Li	< 4	0	0	-	-	Li	< 4	-		-	-
Mg	851.0	7.70	0.90	_	_	Mg	819.0	2.80	0.34	_	-
Mn	0.19	0.01	7.37	-		Mn	0.33	0.06	16.97	_	-
Mo	0.50	0.14	28.20	0.63	0.03	Mo	0.30	-	-	0.43	0.02
Na	2680.0	21.0	0.78	-	-	Na	2675.0	7.10	0.26	-	-
Νί	< 0.4	0	0	_	_	Ni	< 0.4	_	_	_	_
P	1465.0	5.80	0.39	_	-	P	1475.0	7.10	0.48		_
S	2613.0	9.60	0.37	-		S	2595.0	21	0.81	_	_
Sb	< 2	0	0	3.70	0.19	Sb	2.50	0.71	28.40	3.10	0.16
Se	< 3	0	0	3.50	0.18	Se	< 3			3.30	0.17
Sn	7.00	4.50	64.29	-	-	Sn	4.90	0.28	5.71	-	-
Sr	0.01	0	0	_	_	Sr	0.01	_	_	_	_
V	0.27	0.10	35.19	_	_	V	0.30	0.14	47.10	-	_
W	< 1.25	0.50	40.0	2.50	0.13	W	6.0	1.41	23.50	8.20	0.41
Zn	32.20	0.30	0.93	34.30	1.71	Zn	29.95	0.21	0.71	31.24	1.56

Notes: the unit in Table 5-Table 6 is ppm

Table 7	Element cor	1centratio	ns of Megy	F.M.T m	Table 8	Element concentrations of Megy F. M. milk power					
Element	ICP-OES	SD	RSD	INAA	SD	Element	ICP-OES	SD	RSD	INAA	SD
Al	< 2	0	0		-	AJ	< 1.85	0.49	26.70		
В	2.0	0.59	29.50	-	-	В	4.25	0.07	1.65	-	-
Ba	0.27	0.01	1.87	-	-	Ba	0.15	0.04	24.14	-	-
Ca	4375.0	40.0	0.91	4420.0	132,60	Ca	4495.0	21.0	0.47	4722.0	141.66
Ca	< 0.2	0	0	0.32	0.02	Co	< 0.2	0	0	0.32	0.02
Cr	< 0.2	0	0	0.35	0.02	$\mathbf{c_r}$	< 0.2	0	0	0.28	0.01
Cu	2.80	0.21	7.50	-		Cu	3.80	0.28	7.47	_	-
Fe	68.60	0.70	1.02	70.20	3.51	Fe	14.0	0	0	16.70	0.84
K	8510.0	69.0	0.81	_	-	K	8390.0	0	0	-	_
Li	< 4	0	0	_	_	Li	< 4	0	0	-	_
Mg	400.0	4.30	1.08	-	_	Mg	398.50	0.71	0.18	_	_
Mn	0.08	0.01	17.8	_	_	Mn	0.15	0.01	9.33	_	-
Mo	0.43	0.19	45	0.52	0.03	Mo	< 0.3	0	0	0.42	0.02
Na	1370.0	12.0	0.89	_	_	Na	1245.0	7.10	0.57	_	_
Ni	< 0.4	0	0	_	_	Ni	0.4	0	0	-	_
P	528.0	5.70	1.08	_	_	Р	643.0	6.40	0.99	_	-
S	1660.0	18.0	1.08	_	_	S	1260.0	14.0	1.11	-	_
Sb	< 2	0	0	1.20	0.06	Sb	< 2	0	0	3.40	0.17
Se	< 3	0	0	4.30	0.22	Se	< 3	0	0	4.30	0.22
Sn	4.80	0.28	5.83	_	_	Sn	4.90	0.99	20.20	_	_
Sr	0.54	0.01	1.77	-	-	Sr	0.01	0	0	_	_
٧	< 0.2	0	0	_	_	V	< 0.20	0	0	_	_
W	3	1.83	60.86	6.30	0.32	W	< 5.0	1.41	28.20	6.10	0.31
Zn	30.30	0.23	0.76	32.50	1.63	Zn	39.20	0.14	0.36	30.51	1.49

3 **Conclusions**

The results reveal that the major and trace element find in the all types of milk powder are play an important part in grow of human body as in general. So the effect of these elements are in building of bone and teeth. They consist of body cells, blood and muscles also they chare in protein structure. From these results its clear that the important of milk to human body, recommended that all people for all age must drink at least one cup of milk daily.

Its evedains from these results that their exisit a very low percentage of toxic elements Sb and Zn. These toxic elements come from the fertilizer and pesticides. We recommend avoiding of spraying pesticides on the plants which would be used for nutrients of animals, to get milk free of toxic elements. Also, their exist a good agreement Notes: the unit in Table 7-9 is ppm between the results of the two methods of analysis,

Table 9 Element concentrations of Nstogeen milk powder

Element	ICP-OES	SĐ	RSD	INAA	SD
Al	1.33	0.43	32.38		_
В	1.0	0.28	28.0	-	-
Ba	0.11	0.02	20.91	-	-
Ca	4051.0	54.0	1.33	4420.0	132.60
Co	< 0.2	0	0	0.31	0.02
Cr	< 0.2	0	0	0.26	0.01
Cu	3.36	0.22	6.47	_	_
Fe	65.0	1.10	1.69	70.21	3.51
K	9097.1	110.0	1.21	-	-
Li	< 4	0	0	-	-
Mg	376.70	5.40	1.43	-	-
Mn	0.51	0.02	3.14	=	-
Mo	< 0.3	0	0	0.42	0.02
Na	1198.57	17.0	1.42	-	-
Ni	< 0.4	0	0	-	-
P	469.0	7.20	1.54	-	-
S	1555.71	24.0	1.54	=-	-
Sb	< 2	0	0	3.20	0.16
Se	< 3	0	0	4.10	0.21
Sn	3.98	0.67	16.75	-	_
Sr	0.01	0	0	-	-
V	< 0.2	0.04	17.64	-	-
W	< 7.14	2.19	30.67	8.40	0.42
Zn	41.20	0.93	2.26	43.50	2.18

ICP and NAA, validating the accuracy of the present works.

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