

# Research article

# Analysis of arsenic in infant rice cereals by Inductively Coupled Plasma Mass Spectrometry

# Martina Esteria\*, Siti Morin Sinaga, Effendy De Lux Putra

Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Universitas Sumatera Utara, Padang Bulan, Medan, 20155, Indonesia.

Key words: Arsenic, Infant rice cereals, Baby Rice, Inductively Coupled Plasma-Mass Spectrometry.

\*Corresponding Author: Martina Esteria, Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Universitas Sumatera Utara, Padang Bulan, Medan, 20155, Indonesia.

#### Abstract

**Objective:** The aim of this research was to determine arsenic concentration in infant rice cereals and to find out whether arsenic levels from infant rice cereals still meet the standards set by the National Agency of Drug and Food Control of Republic of Indonesia (NADFC). **Method:** The arsenic concentration was determined with an ICP-MS method and the analytical method was validated by measuring accuracy, precision, detection limits and quantitation limits. **Result:** The results of the validation method were obtained recoveries of spiked sample 96.06%, RSD 2.33%, limit detection and limit quantification 0.24  $\mu$ g kg<sup>-1</sup> and 0.78  $\mu$ g kg<sup>1</sup>. The arsenic levels in infant rice cereals were in the range 12.30 – 106.15  $\mu$ g kg<sup>-1</sup> (n=12), on average they were 35.57  $\mu$ g kg<sup>-1</sup>. **Conclusion:** Arsenic concentration in infant rice cereals maximum 20  $\mu$ g/kg, there are nine samples whose arsenic levels exceed the requirements.

#### Introduction

Arsenic is an element in the earth's crust and is very small in water, soil and air from volcanic eruptions and industrial activities such as mining, smelting and burning coal. Arsenic is also used as wood preservatives, pesticides, fertilizers, additives for animal feed and as medicinal ingredients [1].

Arsenic is a metal that belongs to a group of human carcinogens that cause cancer of the skin, kidneys, bladder and lungs [2]. Chronic effects of arsenic exposure cause skin lesions, neurotoxicity, cardiovascular disease, diabetes and cancer [3].

Babies are a population group that is most vulnerable to the toxic effects of arsenic due to higher absorption of arsenic in the digestive tract, faster metabolic processes, detoxification systems that are not fully developed and more food consumption when compared to body weight. [4].

Rice is a staple food that is widely consumed by people in the world and it is also a food source that is often exposed to arsenic. Rice exposed to arsenic comes from the use of pesticides, fertilizers and soil and irrigation water contaminated by arsenic [5].

Generally, mothers introduced rice cereal as the first solid food for infants aged 4-6 months [6]. Arsenic exposure to food is estimated to be about three times higher in infants than in adults, because intake per weight is higher in infants, in addition to less infant food diversity than adults [7].

# Materials and methods

Samples of infant rice cereals (n=12) obtained from baby shop or supermarket in Indonesia. The sampling method was carried out purposively on the consideration that the most material composition in the sample was made from rice and each brand was represented by one sample.

#### Chemicals

Ultrapure water (resistivity 18 M $\Omega$  cm<sup>-1</sup>) from a Milipore water purification system was used throughout this study. Suprapur grade HNO<sub>3</sub> (65%) and H<sub>2</sub>O<sub>2</sub> (30%) were purchased from Merck. Arsenic standard solutions grade ICP-MS (1000 µg mL<sup>-1</sup>) was obtained from Merck (Germany).

#### Instrumentation

All of the measurements were conducted using an ICP-MS (Agilent 7800) instrument with PeriPump sample introduction, MicroMist nebulizer, x-lens ion lens, Scotttype double-pass spray chamber and helium collision mode. Standard, blank and sample solutions were delivered using autosampler. Instrument performance was checked using tuning solution containing with 1  $\mu$ g L<sup>-1</sup> Ce, Co, Li, Mg, Tl, Y (Agilent). Based on the instrument manufacturer, the following parameters were checked during the daily performance analysis: sensitivity (Li, Y and Tl), doubly charges (Co) and oxides (Ce) ratio. ICP-MS Operating conditions are shown in Table 1.

Table 1. ICP-MS operating conditions.

Operating Conditions	Value
ICP Rf power, W	1550
Sample depth, mm	8.0
Nebulizer Gas, L min-1	1.04
Auxiliary Gas, L min <sup>-1</sup>	0.90
Plasma Gas, L min-1	15.0
Helium flow rate, L min-1	4.0
Integration time, sec	0.1
Monitored Masses	$^{75}$ As
Sampler/skimmer cones	Nickel

#### Sample preparation

The solid samples were homogenized by blender to become a fine powder. The powdered samples were weighed accurately to a weight of 0.5 g into a microwave vessel, added with 5 mL of 65% HNO<sub>3</sub> and 1 mL of 30% H<sub>2</sub>O<sub>2</sub>. Then let stand for 10 minutes so the sample dissolves. The vessel is put into the microwave (CEM MARS 6) at 200°C using 4 stage program: to 100°C in 5 minutes, to 130°C in 5 minutes, to 160°C in 5 minutes, to 200°C in 7 minutes and held for 10 minutes and cooling down to 80°C. Samples were cooled to room temperature and transferred into a volumetric flask 25 mL and the vessel is rinsed with ultrapure water up to 25 mL. The sample solution was analyzed for arsenic concentration using Inductively Coupled Plasma-Mass Spectrometer at m/z 75.

## Method validation

Method validation was performed with parameters of linearity, accuracy, precision, detection limit and quantitation limit. Linearity was obtained by preparing a calibration curve from several concentrations of the standard solution and the calculation of the correlation coefficient (r). Accuracy was established by adding standard solution method, and then proceeds with sample analysis procedure as previously done using Inductively Coupled Plasma-Mass Spectrometer at m/z 75, while precision was measured as relative standard deviation. Determination of detection limits and quantitation limits based on the calculation of the calibration curve obtained.

# Results

From the validation study, it can be showed that excellent trueness (% recovery) and good precision were obtained. The detection limit and quantification limit achieved were low enough and suitable for determining arsenic in infant rice cereal at the low levels found in the samples. Method validation performed are shown in table 2.

Table 2. Method validation performed.				
Parameters	Arsenic			
Working range (ng mL <sup>-1</sup> )	0.0-5.0			
Correlation coefficient, r	0.9993			
Recovery (%)	96.0624			
RSD (%)	2.3278			
LOD ( $\mu g k g^{-1}$ )	0.2353			
LOQ (µg kg <sup>-1</sup> )	0.7844			

Table 3. Result of arsenic analysis in infant rice cereal using Inductively Coupled Plasma-Mass Spectrometer.

Sample	Product Description	Made In	Intensity (CPS)	Concentration (µg kg <sup>-1</sup> )	Mean Value	Standard Deviation
A	Rice cereal	Indonesia	3501.7111	31.9290	31.6503	0.3500
			3491.6944	31.9514		
			3925.1711	31.3476		
			3838.4711	31.1924		
			3565.0611	31.4893		
			3598.4178	31.9922		
В	Rice cereal	Indonesia	2668.1678	23.4757	23.4740	0.1996
			2938.2078	23.8304		
			2788.1844	23.4498		
			2664.8011	23.4435		
			2604.8111	23.4335		
			2808.1811	23.2113		
С	Rice cereal	Malaysia	5105.6278	48.0870	48.3730	0.2388
		-	5459.1044	48.7434		
			5339.0111	48.3472		
			5342.3778	48.2036		
			5342.3444	48.3046		

			5339.0378	48.5519		
D	Rice cereal	Indonesia	1974.6711	17.1412	17.2678	0.3060
		muonestu	2178.0444	17.2018	17.2070	0.5000
			2031.3478	17.4692		
			2098.0244	17.7336		
			1924.6578	16.8333		
			2044.6844	17.2275		
E	Rice single grain	USA	11319.5778	106.8816	106.1521	0.7227
	cereal		11029.2611	106.4624		
			11256.1478	106.3808		
			11536.4111	105.2012		
			11523.0344	105.2948		
			11052.6744	106.6917		
7	Rice cereal	England	5649.1344	52.9678	53.3029	0.4705
		e	5669.1844	53.4488		
			5555.7678	52.5879		
			5632.5078	53.2587		
			5946.0111	53.8495		
			5669.1478	53.7047		
Ĵ	Mixed grain cereal	England	4518.7144	41.7068	42.2856	0.5729
	-	-	4748.7878	42.8584		
			4728.7778	42.5061		
			4582.0544	42.7157		
			4935.5278	42.4812		
			4432.0444	41.4453		
ł	Baby rice	Indonesia	2248.0578	18.5381	18.6974	0.1584
	-		2294.7378	18.8767		
			2304.7311	18.7090		
			2434.7744	18.8986		
			2178.0378	18.5909		
			2101.3611	18.5711		
	Organic baby rice	Indonesia	3538.3744	31.0783	32.1317	0.6746
			3661.7544	32.6541		
			3655.1011	31.6065		
			3695.0944	32.7122		
			3531.6844	32.6582		
			3475.0311	32.0809		
	Organic baby rice	Indonesia	1637.9544	12.2366	12.3011	0.3347
			1437.9111	12.0338		
			1521.2644	12.8244		
			1564.6144	12.0361		
			1444.5878	12.0755		
		1501.2644	12.6001			
Κ	Organic baby rice	Indonesia	7339.9644	68.6526	67.9254	0.7434
			7213.2711	68.9687		
			7063.1944	67.4085		
			7086.5511	67.6519		
			7099.8811	67.8374		
			7033.2244	67.0331		
L Organic baby rice	Organic baby rice	Indonesia	2838.2044	21.9303	21.6829	0.2040
			2688.1511	21.6102		
			2388.0644	21.3790		
			2511.4478	21.8308		
			2758.1644	21.5570		
		2611.4544	21.7898			

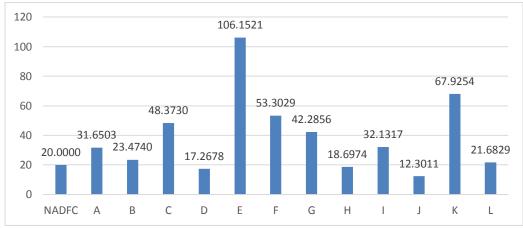


Figure 1. Arsenic level in infant rice cereal and NADFC requirements.

#### Discussion

Table 3 shows that infant rice cereal contains different concentration of arsenic for each sample. The safety of infant rice cereal in this study refers to the arsenic safe limit stipulated by the regulation of the NADFC in 2017 about the maximum limit of heavy metal contamination in processed foods which is not more than 20 µg kg<sup>-1</sup>[8].

Based on Figure 1, a total of 12 infant rice cereal samples were tested, there was only three sample whose arsenic concentration met the NADFC requirements, it was D, H and J with arsenic levels of 17.2678; 18.6974; 12.3011  $\mu$ g kg<sup>-1</sup> and there were nine samples whose arsenic levels exceeded the NADFC requirements, that is between 21.6829  $\mu$ g kg<sup>-1</sup> to 106.1521  $\mu$ g kg<sup>-1</sup>. Sample E with the highest arsenic concentration which reached five times the required by the NADFC.

Based on Table 3, arsenic concentration produced in USA are higher than those produced in Indonesia. Arsenic levels in rice and rice-based products vary due to species and genetic differences from rice, arsenic concentrations in soil and irrigation water, rice planting methods and history of the use of pesticides and chemical fertilizers on agricultural land [9, 10]. Geographical factors of soil can also cause arsenic levels in baby food products derived from rice to vary [11]. The results of research by Williams, *et al.*, (2005) reported that arsenic levels in rice originating from India were lower than rice originating from Bangladesh, Europe, America. Average arsenic levels in rice from Bangladesh was 0.13  $\mu$ g/g; rice from Europe was 0.18  $\mu$ g/g; rice from America was 0.26  $\mu$ g/g [12].

Rice-based baby foods are likely to contain arsenic derived from the composition of ingredients or because of contamination during food processing [2]. When it is stated that rice flour is the most important and dominant composition in baby food, arsenic levels in the sample are higher than baby food mixed with other food ingredients [13].

#### Conclusion

In general, infant rice cereals are widely used during weaning young children because it is easy to get, the price is cheap, bland taste, nutritional value and low allergic potential. Different trademarks affect arsenic levels in baby rice cereals because it is related to the amount of rice in the product, rice origin and manufacturing process. The results of this study showed that 9 of the 12 infant rice cereal samples tested were higher than the NADFC maximum level. Our study suggests that cereal makers need to take immediate steps to reduce arsenic in their infant rice cereal. This action is especially needed to protect infants who rely on rice-based cereal as a staple food.

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