

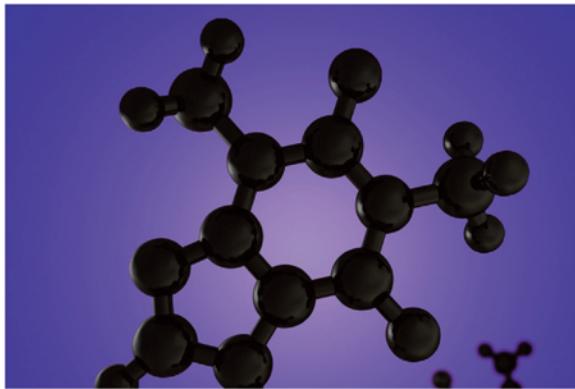


Food Safety Action Plan

REPORT

2009-2010 Targeted Surveys

Chemistry



Arsenic Speciation in Rice and Pear Products

TS-CHEM-09/10-02

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1. Executive Summary

The Food Safety Action Plan (FSAP) aims to modernize and enhance Canada's food safety system. The FSAP unites multiple partners in ensuring safe foods for Canadians.

Arsenic is a naturally occurring element found in trace amounts in rock, soil, water and air. Primary routes of human exposure to arsenic are through drinking water and food. The arsenic in food and water are generally considered to be from normal accumulation from the environment. Arsenic can exist in both organic (elemental arsenic combined with carbon) and inorganic forms (elemental arsenic coupled with oxygen, chlorine, sulphur or other elements other than carbon) in food, with the inorganic forms being the more toxic form. The ratio of arsenic species (organic and inorganic) can vary widely depending on the source of contamination and the commodities in which it is present. Inorganic arsenic is generally found at very low levels in foods, and is the predominant species in drinking water and vegetables (including rice), whereas organic forms are generally detected at higher levels and are the predominant species found in aquatic organisms (including fish and seafood). Chronic exposure to arsenic may lead to a variety of detrimental health effects in humans, most notably cancer.

The main objectives of this survey were:

- To provide baseline surveillance data for total arsenic levels in rice and pear products.
- To examine the proportions of inorganic arsenic in rice in pear products with a new analytical method.

A total of 213 samples were collected (108 pear products and 105 rice products). Both the rice and the pear products originated from domestic and imported origins. Samples were tested "as sold" to determine the total arsenic content. Those samples found to contain measurable levels of arsenic were further tested to determine the levels of various organic and inorganic species present.

All rice samples analyzed contained detectable levels of total arsenic. Brown rice contained the highest average levels of total arsenic at 0.24 ppm, followed by white rice at 0.14 ppm, rice drinks at 0.02 ppm and sake (a rice based alcoholic beverage) at 0.01 ppm. Arsenic speciation results indicate that the majority of the arsenic species present in white and brown rice samples tested are inorganic in nature, and that rice drinks contain a higher proportion of organic species. On average, inorganic arsenic species comprised 70-80% of all arsenic species detected in white and brown rice. In rice drinks and sake, inorganic arsenic accounted for an average of approximately 65% and 48% of all arsenic species detected, respectively. There are currently no established maximum levels for total arsenic or specific arsenic species in rice commodities. The levels of arsenic detected in the rice commodities tested herein would not be expected to pose a human health risk to the Canadian public.

With the exception of three products, all pear samples analyzed contained detectable levels of total arsenic. Pear snacks contained the highest levels of total arsenic with an

average total arsenic concentration of 0.036 ppm, followed by pear juice at 0.007 ppm and pear nectar and baby food, which both had 0.003 ppm. Speciation results indicated that the pear juice contained a lower proportion of inorganic arsenic species, with approximately 20% of all arsenic species detected being inorganic in nature. Pear snacks, pear nectar and pear baby food had slightly higher proportions of inorganic arsenic detected, with about 35-55% of the total arsenic species being inorganic in nature. There is a maximum tolerance in Canada for arsenic in fruit juices, fruit nectar, and ready-to-serve beverages of 0.1 ppm. This would include pear juices and nectars, which were analyzed in this survey. All juice samples analyzed were well below this maximum tolerance. Levels of arsenic detected in other pear commodities would not be expected to pose an unacceptable human health risk to the Canadian consumer.

2. Introduction

2.1. Food Safety Action Plan

The Food Safety Action Plan (FSAP) aims to modernize and enhance Canada's food safety system. The FSAP unites multiple partners in ensuring safe foods for Canadians.

Within FSAP, the Canadian Food Inspection Agency (CFIA) gained increased capacity to monitor potential food risks and to prevent unsafe food products from entering the Canadian marketplace. The CFIA fulfils this mandate through an enhanced surveillance initiative, which includes targeted surveys. The CFIA works on these initiatives with input from Federal partners, including Agriculture Agri-Food Canada and Health Canada, as well as Provincial and Territorial (P/T) representatives.

2.2. Targeted Surveys

Targeted surveys are pilot studies used to gather information regarding the potential levels of chemical residues in defined commodities. The surveys are designed to answer specific questions; therefore, unlike monitoring activities, testing of a particular chemical hazard is targeted to commodity types and/or geographical areas. Due to the vast number of chemical hazards and food commodity combinations, it is not possible, nor should it be necessary, to use targeted surveys to identify and quantify all chemical hazards in foods.

To identify food-hazard combinations of greatest potential health risk, the CFIA uses a combination of media reports, scientific literature and/or a risk-based model developed by the Food Safety Science Committee (FSSC).

In March of 2008, the CFIA issued a voluntary recall of pear juice marketed to toddlers due to higher than normal arsenic levels¹. At the time the CFIA Laboratories did not have a method available to distinguish the more toxic forms of inorganic arsenic from total arsenic levels. With the development of this methodology a targeted survey exploring the levels of both total and inorganic arsenic in pear products would enable a more refined human dietary assessment.

There have been a number of studies that have indicated that elevated levels of inorganic arsenic in rice may contribute significantly to the inorganic arsenic dietary intake^{4,9}. Rice is particularly susceptible to arsenic contamination, as it is generally grown under flooded conditions where arsenic mobility is high. Again, since a new methodology has allowed for the quantification of arsenic species, investigation of the levels of total and inorganic arsenic in rice and rice drinks would be valuable.

2.3. Acts and Regulations Relating to Arsenic

2.3.1. Canadian Regulations

The *Canadian Food Inspection Agency Act* stipulates that the CFIA is responsible for enforcing restrictions on the production, sale, composition and content of foods and food products as outlined in the *Food and Drugs Act & Regulations* (FDAR).

Health Canada (HC) determines the legal limits of contaminants and pesticide residues in food. Tolerances are established to be used as a risk management tool and generally only for foods that significantly contribute to the total dietary exposure. Currently HC has established a maximum allowable concentration of arsenic in drinking water at 0.010 ppm. There are, at present, three tolerances established in the FDAR (Section B.15.001- Table I) for arsenic in foodstuffs:

1. fish protein (3.5 ppm)
2. edible bone meal (1 ppm)
3. fruit juices, fruit nectar, beverages when ready to serve and water in sealed containers other than mineral water or spring water (0.1 ppm)

There are also Canadian tolerances in animal tissues and eggs stemming from the use of veterinary drugs that contain arsenic (Section B.15.001- Table III).

Foods for which tolerances have not been established will still contain low levels of arsenic. However, tolerances have not been established because they present a low health risk to the general Canadian population. Despite the limited number of general tolerances for arsenic in food, higher than average levels of arsenic in specific foods may still be assessed by HC. Health Canada assesses any findings of elevated levels of arsenic in food on a case-by-case basis using the most current scientific data available to assess if there is an unacceptable risk posed by the consumption of a certain commodity. When levels of arsenic in food are deemed to be unsafe, corrective actions such as public recalls, product retention and/or the establishment of maximum limits are actioned by the CFIA and HC.

Targeted surveys may be used to identify emerging food-hazard issues. In such cases, pertinent maximum limits may be lacking. Thus, results from targeted surveys can provide baseline data which may be used to perform health risk assessment, and subsequently establish maximum limits.

2.3.2. International Regulations

The US Food and Drug Administration (FDA) has standards for inorganic arsenic ranging from 0.5-2 ppm in animal tissues and eggs resulting from the treatment of animals with veterinary drugs². In the UK, a level of 1 ppm arsenic is set for all foods; however this standard was established in 1959, prior to the confirmation of inorganic arsenic as a chronic carcinogen by the International Agency for Research on Cancer (IARC)³. China has a food standard limit of 0.150 ppm inorganic arsenic⁴. Codex Alimentarius has set a number of standards for arsenic (natural mineral water, edible fats and oils, fat spreads and blended spreads, certain animal fats, olive oils, vegetable oils and food grade salts)⁵.

2.4. Analytical Testing

In order to address potential hazards in the food supply, CFIA laboratories develop and validate new analytical methods. These methods may provide increased sensitivity of detection, multi-analysis detection, or analysis of existing hazards in new matrices. To become accredited, an analytical method must meet validation parameters pertaining to recovery, selectivity, specificity, accuracy, linearity, range, precision, repeatability or reproducibility, limit of quantification (LOQ), and limit of detection (LOD).

Samples from the arsenic speciation targeted surveys were analyzed by the CFIA's Dartmouth, Nova Scotia laboratory. Samples were tested "as sold" (i.e uncooked form) to determine the total arsenic content. Those samples found to contain levels of arsenic which could be speciated were further tested to determine the levels of various organic and inorganic species present.

Total Metals Screen

Samples for the arsenic speciation survey were initially analyzed using a total metals screen methodology, which is meant to be a rapid assessment of 12 different metals including arsenic, but might lack some of the specificity of the speciation method detailed below. This method applied microwave assisted digestion and Inductively Coupled Plasma Mass Spectrometry (ICP-MS). If samples contained enough arsenic to warrant speciation, a second method was applied in order to quantify the arsenic species present (see below).

Arsenic Speciation Method

This method is more labour intensive and provides a means of determining inorganic arsenic (in the form of arsenite and arsenate) and organic arsenic (arsenobetaine, arsenocholine, mono methyl arsenic disodium salt and cacodylic acid) in food by Gas Chromatography- Inductively Coupled Plasma Mass Spectrometry (GC-ICP-MS) (see Table 1). The LOD for rice and pear products are presented in Table 2. The LOQ was calculated as 3 times the LOD for each matrix. It should be noted that the arsenic speciation method analyzed for the specific suite of arsenic species mentioned above. This may not have captured all arsenic species present.

TABLE 1. Arsenic species examined in the current targeted survey.

Species	Form	Synonym	Chemical formula	Relative Toxicity
AsIII	Inorganic	As ³⁺ , Arsenious acid, Arsenite	As(OH) ₃	Highly toxic
AsV	Inorganic	As ⁵⁺ , Arsenic acid, Arsenate	H ₃ AsO ₄	Highly toxic
AsC	Organic	Arsenocholine	C ₅ H ₁₄ AsBrO	Virtually non-toxic
AsB	Organic	Arsenobetaine	C ₅ H ₁₁ AsO ₂	Virtually non-toxic
MMA	Organic	Mono Methyl Arsonic Disodium Salt	CH ₃ AsO ₃ Na	Less toxic
DMA	Organic	Dimethyl Arsenic Acid, Cacodylic acid	C ₂ H ₇ AsO ₂	Less toxic

TABLE 2. Analytical method particulars.

Arsenic Species	LOD (ppb) in Rice Matrices	LOD (ppb) in Pear Matrices (excluding juice)	LOD (ppb) in Pear Juice
Total Arsenic	1	1	1
AsC	0.39	0.52	0.21
AsB	0.43	0.24	0.10
AsIII	0.68	0.66	0.26
DMA	0.71	0.37	0.15
MMA	0.98	0.58	0.23
AsV	4.80	2.67	0.83

3. Survey Samples & Analytical Methods

3.1. Rationale

3.1.1. Arsenic Overview

Arsenic is an element that naturally occurs in the earth's crust. Although arsenic can be found in its elemental form (As), it is more commonly found in combination with other elements. Inorganic arsenic compounds are formed when arsenic is combined with oxygen, chlorine and sulphur which naturally occur in soils and rocks. Organic arsenic compounds are formed when arsenic is conjugated with carbon and hydrogen which may occur as the result of plant or animal metabolism.

In the past inorganic arsenic compounds have been used as a wood preservative and as components of agricultural chemicals, however the practice of using inorganic arsenic compounds for agricultural use and for the treatment of wood destined for residential uses have been discontinued in Canada due to health concerns. Organic arsenic compounds are used as pesticide components and some compounds are used as additives in animal feed. They also are used in automobile batteries, in semiconductors and in light-emitting diodes².

Arsenic in the environment may be deposited in the air, water and soil via wind distribution, or may be deposited in water via runoff or leaching. It may be absorbed by humans via ingestion of contaminated food and/or water or through inhalation of contaminated dusts.

The majority of human exposure to total arsenic is through contaminated drinking water and food⁶. Total arsenic intake for a typical North American adult has been estimated to be approximately 50 µg/day, with inorganic arsenic comprising 20-40% of the total dietary arsenic intake⁷.

Generally speaking, inorganic arsenic species (i.e AsIII and AsV) exert greater toxicity than organic arsenic species (AsB, AsC, DMA, MMA)^{5,8}. Most cases of human toxicity from arsenic have been associated with exposure to inorganic arsenic. Besides being a potent human carcinogen, oral exposure to the inorganic form may result in dermal, cardiovascular, respiratory, gastrointestinal, haematological, hepatic, and neurological effects⁵. Animal data has also suggested that oral exposure to inorganic arsenic may lead to increased risk of reproductive issues. In the case of organic arsenic, the gastrointestinal tract, the renal and urinary systems appears to be the most sensitive targets in humans⁵.

3.1.2. Arsenic in Rice and Pear Products

Cereals and rice have been identified as significant sources of both total arsenic and inorganic arsenic (AsIII and AsV)^{5,9}. Concerns over the levels of inorganic arsenic contamination in rice have been growing for a variety of reasons; one of them being that rice is a heavily consumed food commodity. Rice accounts for nearly 50% of the grain consumption in the world⁴. In Canada, rice available for consumption reached 7.0 kg per person in 2008¹⁰. Rice is particularly susceptible to arsenic accumulation compared to other cereals, as it is generally grown under flooded conditions where arsenic mobility is high. Groundwater contamination by arsenic has been reported in many rice growing countries, most notably in Bangladesh, China and India¹¹. In Bangladesh, a positive correlation between arsenic in groundwater resources, soil and rice has been reported, indicating that food chain contamination takes place because of prolonged irrigation with contaminated water¹¹.

Information on the levels of inorganic arsenic in rice is an important piece of information for risk assessments. But also, as rice products grow in popularity, particularly with

individuals suffering from gluten sensitivities or lactose intolerance, information on levels of inorganic arsenic in processed rice products is imperative.

A health hazard alert was issued by the CFIA on March 11th 2008¹, warning the public that two brands of pear juices for toddlers had elevated levels of arsenic (up to 0.107 ppm). The source of the arsenic in this case was suspected to be the result of pears accumulating arsenic that was present in the soils as a result of historical applications of arsenic-containing pesticides. This survey looks to establish baseline data for the levels of total arsenic and examine the distribution of arsenic species in a variety of pear products including juice, fruit snacks and baby foods.

3.2. Sample Distribution

A total of 213 samples were collected (108 pear products and 105 rice products) (See Figure 1). Both the rice and pear products originated from domestic and imported origins. It is important to note that products labelled as domestic may have been produced with imported ingredients, so no clear conclusions are drawn based on the origins of samples. See Figure 2 for a detailed breakdown of sample origins. The majority of the samples were collected in the Dartmouth, Nova Scotia area, with an additional 50 samples being picked up in the Toronto, Ontario area.

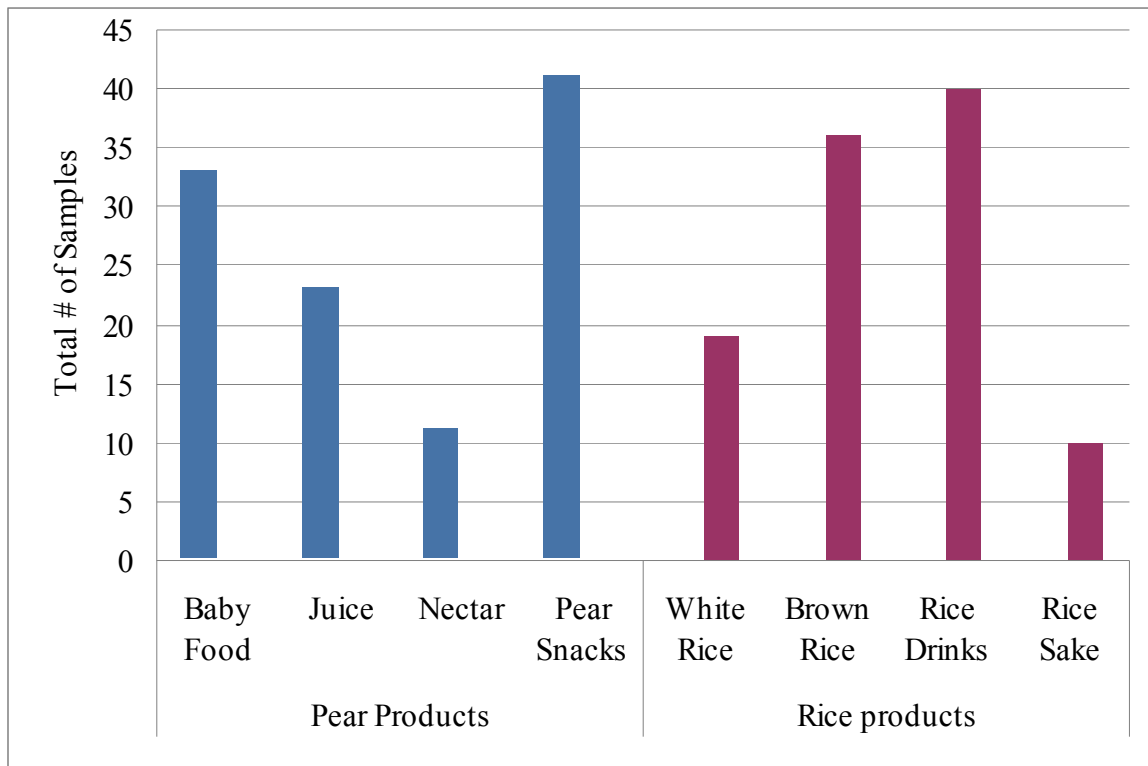


FIGURE 1. Distribution of rice and pear samples by food category

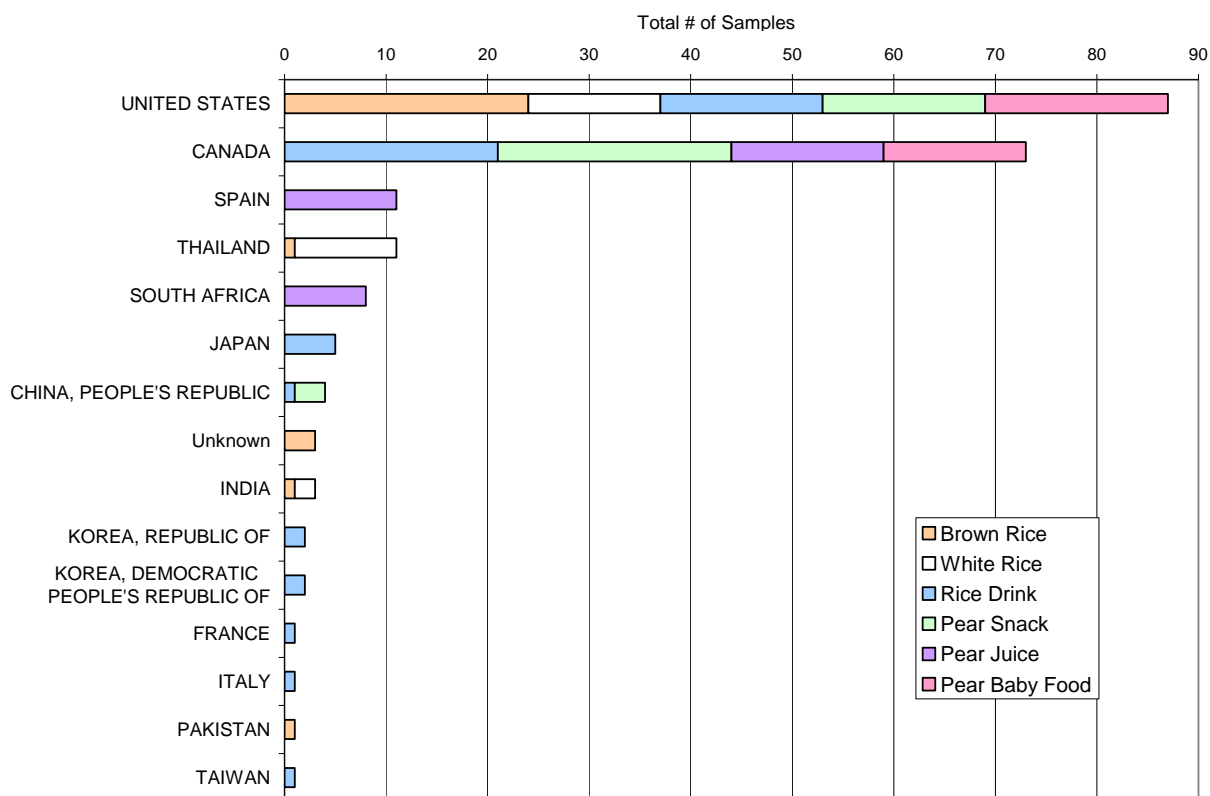


FIGURE 2. Distribution of samples by country of production

3.3. Limitations

A total of 213 samples were collected and analyzed in the 2009-10 arsenic speciation survey. A wide variety of rice and pear products were sampled; they were categorized into the aforementioned categories primarily based on their descriptions. Due to the diverse assortment of products sampled, it is difficult to make comparisons of, or come to any conclusions regarding specific product types or their countries of origin. This data is meant to provide a snapshot of the targeted commodities and has the potential to highlight commodities that warrant further investigation.

4. Results

The results for total arsenic are reported in mg/kg (ppm). The results for the arsenic speciation portion of this survey are reported in $\mu\text{g}/\text{kg}$ (ppb). Two distinct methods were used to generate these two datasets. A general metals screen (Sc) was used to assess the total arsenic concentration and an arsenic speciation method (Sp) was used to quantify the concentrations of six individual arsenic species. Because there were two distinct

analytical methods used, and each method has a level of variability associated with it, it is not possible to directly contrast the results from the two separate methods.

The average total arsenic concentration, as calculated from the total metals screen, for each rice and pear commodity tested is depicted in Figure 3.

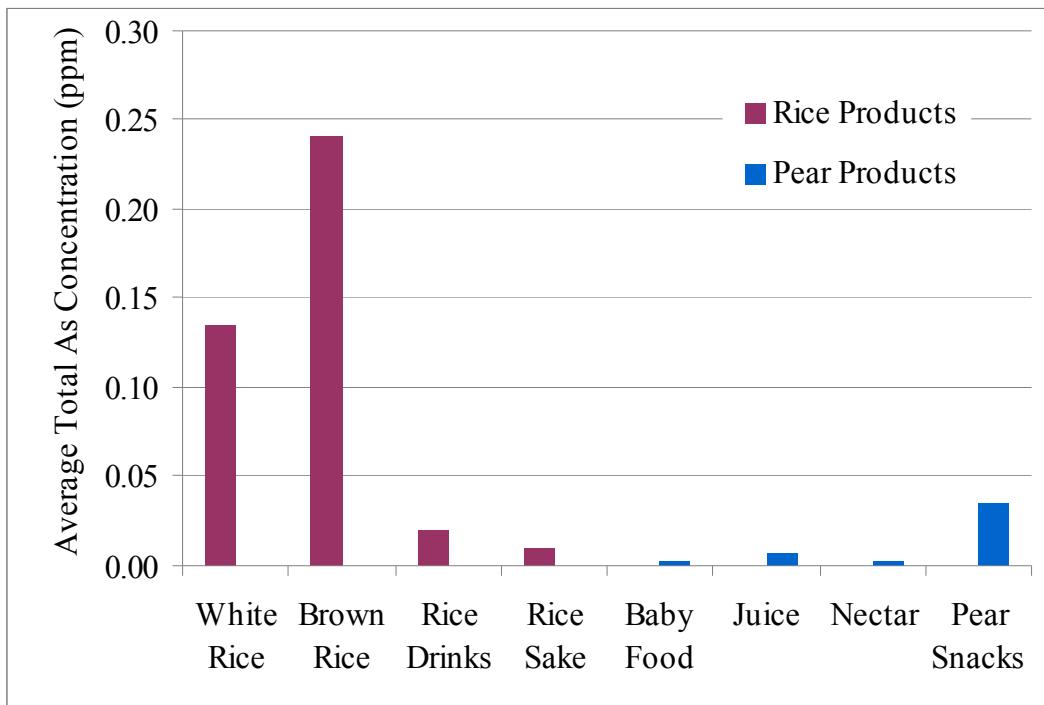


FIGURE 3. Average total arsenic concentration in rice and pear products

4.1. Rice Products

A total of 105 rice products were collected in the 2009/10 Arsenic Targeted Survey and were analyzed according to the protocols described in section 1.4.

Rice samples originated from thirteen countries and a total of four rice product categories were investigated: white rice, brown rice, rice drinks and sake.

A summary of total arsenic levels in rice are presented in Table 3. All rice samples analyzed contained detectable levels of total arsenic. Brown rice contained the highest average levels of total arsenic at 0.241 ppm, followed by white rice at 0.136 ppm, rice drinks at 0.020 ppm and sake at 0.010 ppm.

TABLE 3. Total Arsenic Concentrations in Rice Products (from Sc method)

Rice Category	No. of Samples	Min (ppm)	Max (ppm)	Average (ppm)	Standard Deviation
White Rice	19	0.04	0.190	0.136	0.044
Brown Rice	36	0.05	0.386	0.241	0.087
Rice Drink	40	<LOD*	0.040	0.020	0.009
Sake	10	<LOD*	0.039	0.010	0.010

*Limit of Detection (LOD) for total arsenic = 0.001 ppm

Results from the arsenic speciation method indicate that the majority of the arsenic species present in white and brown rice are inorganic in nature, and that rice drinks contain a higher proportion of organic species. Figure 4 illustrates this fact by presenting the average arsenic species proportion for each of the rice commodities tested. On average inorganic arsenic species (AsIII and AsV) comprised 70-80% of all arsenic species detected in white and brown rice. In rice drinks and sake, inorganic arsenic accounted for approximately 65% and 48% of all arsenic species detected, respectively. This is not unexpected as it is known that, in comparison to seafood, most terrestrial foods contain low total levels of arsenic with a high percentage of the total arsenic being inorganic in nature⁵.

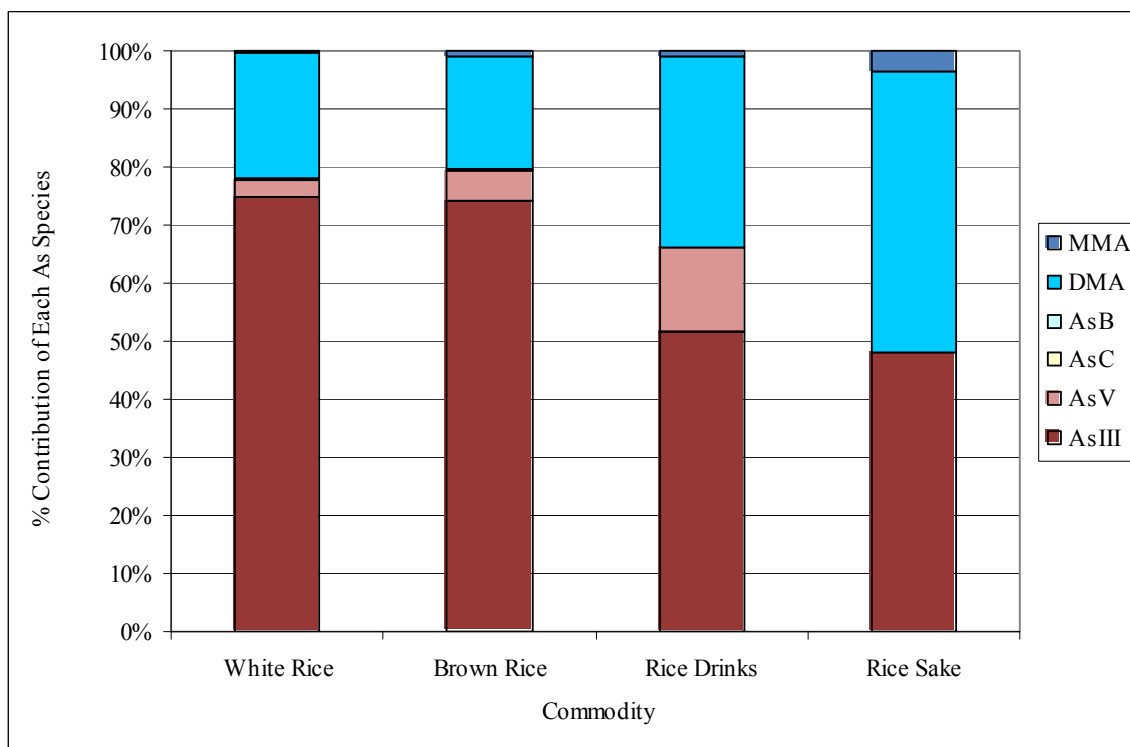


FIGURE 4. Average proportion of arsenic species detected in rice samples (N.B. – red species are inorganic and blue species are organic in nature)

4.2. Pear Products

A total of 108 pear products were collected in the 2009/10 Arsenic Targeted Survey and were analyzed according to the protocols described in section 1.4.

Pear samples originated from five countries and a total of four pear product categories were investigated; these categories included pear snacks (this included fruit leathers, gummy snacks and fruit bars), pear juice, pear nectar (a juice that is not filtered after pulping, resulting in a thicker, more syrupy beverage) and pear baby food.

Total arsenic levels from metals screen for pear products are presented in Table 4. With the exception of three pear products, all samples analyzed contained detectable levels of total arsenic. Pear snacks contained the highest levels of total arsenic with an average total arsenic concentration of 0.036 ppm, followed by pear juice at 0.007 ppm and pear baby food and nectar, which both contained 0.003 ppm.

TABLE 4. Total Arsenic Concentrations in Pear Products (Sc method)

Pear Category	No. of Samples	Min (ppm)	Max (ppm)	Average⁺ (ppm)	Standard Deviation
Pear Snacks	41	0.002	0.091	0.036	0.025
Pear Juice	23	<LOD*	0.026	0.007	0.005
Pear Baby Food	33	<LOD*	0.009	0.003	0.002
Pear Nectar	11	0.002	0.005	0.003	0.001

*Limit of Detection (LOD) for total arsenic = 0.001 ppm

+ Average of samples with detectable levels of total arsenic.

Speciation results indicated that the pear juice contained the lowest proportion of inorganic arsenic species, with approximately 20% of all arsenic species detected being inorganic in nature. Pear snacks, pear nectar and pear baby food had slightly higher proportions of inorganic arsenic detected, with approximately 35%, 45% and 55% of the total arsenic species being inorganic in nature, respectively (Figure 5).

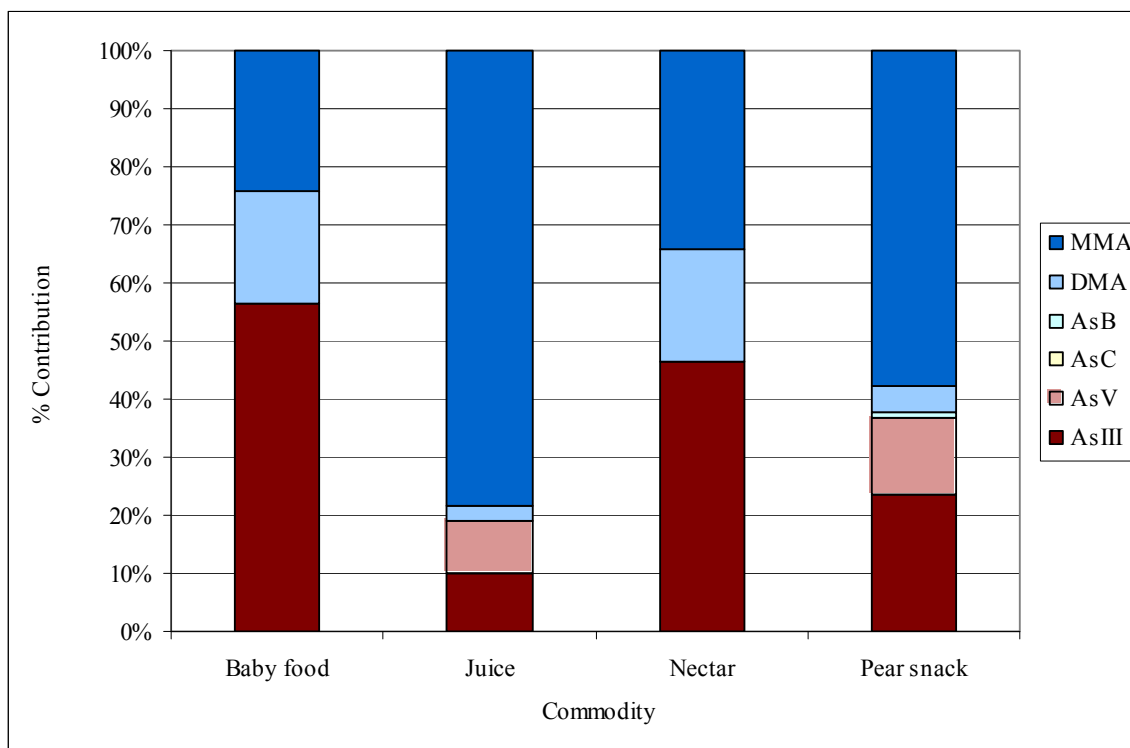


FIGURE 5. Average proportion of arsenic species detected in pear samples (N.B – red species are inorganic and blue species are organic in nature)

5. Discussion and Conclusions

As can be seen in Figure 3, rice products contained greater total arsenic concentrations than pear products. Rice products also displayed a greater proportion of inorganic arsenic species than pear products (Figure 4 and 5). Overall the predominant arsenic species in rice were AsIII and DMA, and in pear were AsIII and MMA. All of these species are known to be mobile in water, which would facilitate uptake of arsenic into the affected plants.

The magnitude and proportions of arsenic species detected in rice and pear products is not unexpected as the source of arsenic contamination in rice is thought to be from contaminated groundwaters, where inorganic arsenic mobility is high¹¹. The source of arsenic contamination in pears is thought to be due to the historical application of arsenic containing pesticides. The arsenic may have become bound to soil components and is gradually taken up by the plants. It should be noted that North America now has restrictions on harmful arsenic based pesticides, and many pear producing regions around the world have discontinued use of inorganic arsenic containing pesticides.

5.1. Comparison of survey results with historical data

5.1.1. Rice

The total arsenic results from the current targeted survey were compared with total arsenic values available from previous surveys conducted by the CFIA as well as concentrations reported by other international organizations.

A variety of rice and rice products were sampled by the CFIA between 2006 and 2010 for the National Chemical Residue Monitoring Program (NCRMP) and the Children's Food Chemical Residue Project. Samples were analyzed for total metals using the total metals screen method detailed in section 1.4. The average concentration of total arsenic in rice grains was 0.145 ppm (n=6), in rice based infant cereals (includes both rice-based cereals and mixed cereals containing rice) the average total arsenic concentration was 0.184 ppm (n=14), and in mixed rice commodities (included snacks and mixed meals) the average total arsenic concentration was 0.200 ppm (n=15). These values are quite similar to the results of this 2009/10 survey where the average total arsenic concentration for all rice products ranged from 0.01 ppm to 0.241 ppm.

Health Canada has analyzed for total arsenic in their Canadian Total Diet Study since 2004. In the 4 samples of rice grain composites analyzed in the course of this study, the average total arsenic level was 0.086 ppm¹². It is important to note that the Total Diet Study samples are analyzed "as consumed" (i.e. have undergone standard food preparation/cooking prior to analysis). This fact, along with the disparity of sample sizes between this survey and the Health Canada reports, make a direct comparison of results not possible.

Similarly, when the results of the current survey are compared with other international studies, both the magnitude of total arsenic detected in rice and rice products, as well as the general proportions of arsenic species detected are in agreement with commonly referenced studies. A study was performed in the United Kingdom in 2009¹³, in which 60 samples of rice drinks were analyzed for both total arsenic and inorganic arsenic. The average total arsenic concentration in rice milks sampled in the UK study was 0.023 ppm and the average inorganic arsenic was 0.012 ppm. These values concur strongly with the data presented in the current study.

A Scientific Opinion on Arsenic in Food was also recently published by the European Food Safety Authority (EFSA)⁵, in which a variety of results on arsenic concentrations in various food commodities were compared. The majority of the data compiled for arsenic reported only total arsenic concentrations. The highest total arsenic concentrations were found in seafood, seaweed and cereal and cereal products, with particularly high concentrations in rice grains and rice-based products. The mean upper bound concentration of total arsenic reported for rice grains and rice based products were 0.142

ppm and 0.166 ppm, respectively, and also reported an inorganic arsenic content that varied between 50 % and 60% of the total arsenic content.

The EFSA scientific opinion concluded that estimated dietary exposures to both the average European consumer, as well as consumers with higher exposure levels left little margin of exposure and the possibility of risk to some consumers could not be excluded. Further to this, it was suggested that dietary exposures to inorganic arsenic be reduced and that in order to refine risk assessments of inorganic arsenic, the production of arsenic speciation data for different food commodities is imperative⁵.

5.1.2. Pear

There is little information on the levels of arsenic (both total and speciated) in pears and pear products. Comparison of the levels of total arsenic found in the current survey with limited data collected by the CFIA for the NCRMP and Children's Food Chemical Residue Project in the past shows that the average total arsenic concentration in pear products (which included fresh pears, canned pears and pear juice) was 0.032 ppm (n=87).

The EFSA Scientific Opinion paper referenced above also reported concentrations of total arsenic in fruits⁵. Although no further breakdown of sample types was given, the mean upper bound concentration of "other fruits" was 0.017 ppm, which is in general agreement with the results reported for pear products analyzed herein.

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