

Canadian Food

# **Food Safety Action Plan**

## REPORT

2009-2010 Targeted Surveys Chemistry



Pesticide residues and metals in dried tea

TS-CHEM-09/10-08



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## **Executive Summary**

The Food Safety Action Plan (FSAP) aims to modernize and enhance Canada's food safety system. As a part of the FSAP enhanced surveillance initiative, targeted surveys are used to test various foods for specific hazards.

There were multiple objectives established for this targeted survey. The first was to provide an initial baseline of surveillance data for the levels of pesticide residues and metals in dried teas available to consumers (i.e., imported and domestically blended). Secondly, to provide the data necessary for the Canadian Food Inspection Agency (CFIA) Calgary Laboratory to validate a new liquid-chromatography mass-spectrometry analytical method for the detection of pesticide residues in dried tea leaves.

Currently federally registered agricultural commodities are monitored by the CFIA under the National Chemical Residue Monitoring Program (NCRMP). The 2009-2010 Pesticide Residues and Metals Survey targeted dried tea, which is not traditionally tested under the CFIA core activities. In total, 100 dried tea samples (24 black, 41 green, 12 herbal/blends, 13 oolong and 10 white teas) were collected from retail stores.

The 100 dried tea samples collected, consisted of loose dried leaves and bagged teas, were analysed for over 340 different pesticide residues and 18 different metals. Thirty-one percent of the samples collected contained no detectable pesticide residues. Of the remaining 69 samples containing detectable pesticide residues, 41 samples contained at least one pesticide residue in violation of the 0.1 ppm General MRL. Oolong tea contained the highest number of pesticide residue violations at 92% of the samples collected (12 out of 13) followed by herbal/blends at 58% (7 out of 12), green tea at 44% (18 out of 41), white tea at 20% (2 out of 10) and black tea at 8% (2 out of 24) of the samples collected per specific tea type. All violations were assessed and appropriate follow-up action was pursued. Exposure to these pesticide residues in tea is not expected to pose a human health concern to consumers given the consumption of tea relative to other food commodities. The overall compliance rate of this targeted survey was 59%.

Of the 18 metals analysed, all metals except beryllium, were detected in all dried tea samples. The levels of metals found in this targeted survey were comparable to the observed levels in dried tea found in the published literature. It should be noted that brewed tea was not analysed in this survey. The results should only be interpreted as dried tea available as sold, and not brewed tea as consumed.

## **1** Introduction

### 1.1 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 targeted surveys with input from other CFIA departments, Health Canada and Provincial and Territorial (P/T) representatives.

### **1.2 Targeted Surveys**

Targeted surveys are pilot surveys used to gather information regarding the potential occurrence 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.

Many agricultural commodities are currently monitored by the CFIA under the National Chemical Residue Monitoring Program (NCRMP) for the presence of pesticide residues and metals. Monitoring is conducted on imported and inter-provincially traded federally regulated commodities (registered) that fall under the <u>Canadian Agricultural Products Act</u> (CAP Act). While the NCRMP tests for pesticide residues and metals in various regulated commodities, its scope is limited to imported and domestically regulated products. Dried tea is a non-federally regulated commodity, therefore routine monitoring at the federal level is limited. The purpose of this targeted survey was to provide an initial baseline of pesticide residue and metals data for dried tea products available to Canadian consumers.

## **1.3 Acts and Regulations Relating to Pesticide Residues and Metals**

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's Pest Management Regulatory Agency (PMRA) is responsible for the registration and regulation of pesticides and the establishment of maximum residue limits (MRLs) under the *Pest Control Products Act* (PCPA). The MRL is the maximum amount of residues that are expected to remain in or on food products when a pesticide is used according to label directions. Currently there are no specific MRLs listed on Health Canada's website for pesticide residues in dried tea leaves. In the absence of a MRL or PMRL, pesticide residues must comply with the Canadian General MRL of 0.1 ppm as stated in the section B.15.002(1) of the *Food and Drug Regulations*.

Maximum levels for chemical contaminants in foods may be expressed as either regulatory tolerances or standards. Regulatory tolerances can be found in Sections B.01.046, B.01.047 and Division 15 of the *Regulations* whereas standards can be viewed on Health Canada's website. There are no Canadian tolerances or standards established for metals in dried tea.

Foods for which maximum levels have not been established may still contain low levels of metals Maximum levels have not been established because these low levels are not expected to pose an unacceptable health risk to the general Canadian population. Despite the limited number of maximum levels for metals in food, higher than average levels of metals in specific foods may still be scrutinized by Health Canada. Health Canada assesses any findings of elevated levels of metals in food on a case-by-case basis using the most current scientific data available to determine if there is a potential health risk. When levels of metals in food are deemed to be unsafe, corrective actions such as public recalls, product retention and/or the establishment of maximum levels are undertaken by the CFIA and Health Canada.

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

Note: Since this survey was completed, Health Canada has established MRLs for fenpropathrin and lambda-cyhalothrin in tea (dried leaves): <u>http://www.hc-sc.gc.ca/cps-spc/pubs/pest/\_decisions/index-eng.php#mrl-lmr</u>

## 2 Pesticide and Metals Survey

#### 2.1 Chemical Hazards in Tea

Some of the world's top tea producing countries are China, Turkey, India, Kenya and Sri Lanka<sup>1</sup>. As this agricultural commodity is a high value cash crop and is susceptible to pest pressures, pesticides can be applied pre-harvest on fresh tea leaves during production and/or post-harvest on dried tea leaves during manufacturing. Different pest pressures and climatic conditions in tea producing areas may result in a potential for pesticides that are not approved or have been banned for use in North America to be used in these areas. The pesticides used in tea production are used for the same reasons they are used in other crops across North America. They are an important tool used in food production because pests such as insects, bacteria, fungi and other organisms can have devastating effects on the quantity and quality of the tea leaves. Although pesticides play an important role in agriculture by protecting food and crops from pests, inappropriate use of pesticides may pose a health risk.

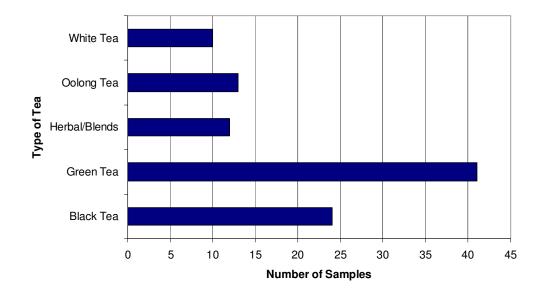
Tea plantations can remain in production for more than 50 years. Throughout this time many applications of fertilizers and pesticides occur, leading to an accumulation of metals and other chemicals in the soil. Due to the physiology of the tea plant, it prefers to grow on acidic soils. Over time soils of a tea plantation become progressively more acidic. High acidity soil favours the disassociation of metals from the soil, making them more available for uptake in the tea plant. The quality of water used in irrigation also contributes to the quality of the soil and in turn the quality of the tea plant.

#### 2.2 Rationale

In Canada, the 2009 per capita consumption of tea was 65.2 L/year which followed coffee (90.0 L/year), beer (77.3 L/year) and soft drinks (71.7 L/year)<sup>2</sup>. Since tea is a highly consumed beverage by the Canadian population, the present targeted survey was designed by the CFIA to gain an appreciation of the levels of pesticide residues and metals in dried tea leaves sold within Canada.

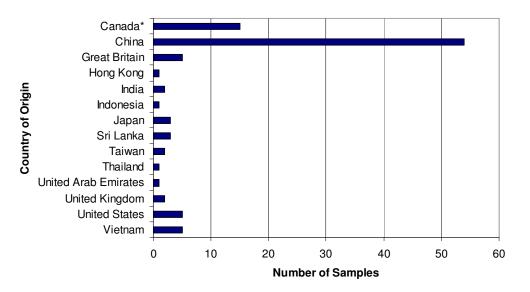
#### 2.3 Sample distribution

The 2009-2010 Pesticide and Metals Survey targeted both loose and bagged tea. A total of 100 samples of black, green, herbal, oolong and white tea were collected in the Calgary area. Teas from Calgary, the fifth largest metropolitan area in Canada, were assumed to generally be representative of all teas in Canada. The CFIA Calgary Laboratory was tasked with validating a new liquid-chromatography mass-spectrometry method for the detection of pesticide residues in dried tea. It is for this reason that samples were only collected in the Calgary area. The distribution of samples by type of commodity is depicted in Figure 2.1. Teas were collected at grocery stores and specialty markets.



#### Figure 2.1. Distribution of sample by tea type.

Dried and bagged teas originated from a total of 13 countries based on market availability. Country of origin is the country of manufacture as stated on the label of the finished product. The determination of country of origin can be hampered by the fact that raw materials are sourced from different countries and are mixed prior to transformation into the final product. As an example, Canada does not have the appropriate climate for growing tea, however Canadian-based companies import raw materials for processing and re-sale into domestic and import markets.



\*Tea is not grown in Canada, rather tea leaves are imported for processing and re-sale into domestic and import markets. When this occurs, products are considered to be a 'Made in Canada from imported ingredients'.

Figure 2.2. Distribution of samples by country of origin.

#### 2.4 Method Details

Tea samples were analysed by an accredited third party laboratory or the Calgary CFIA Laboratory depending on the analytical method used. Third party laboratories are accredited to ISO/IEC 17025, *General Requirements for the Competence of Testing and Calibration Laboratories* (or its replacement by the Standards Council of Canada (SCC)). Sufficient amounts of tea samples were collected to allow for three different analytical methodologies to be conducted on each sample.

#### 2.4.1 Pesticide Analysis

Gas chromatography–mass spectrometry (GC-MS) method entitled "Determination of Pesticides in Tea (by Modified QuEChERS Extraction and GC-MSD Detection (PMR-010-V1.0)" was recently validated by CFIA laboratories. The method can measure approximately 200 pesticide analytes with an analytical range of 0.083 ppm to 0.83 ppm. Please refer to appendix A for a detailed list of pesticide residues analysed. The limit of detection (LOD) for Iprodione, Fenvalerate, Deltamethrin, Chlorthiamid and Chlorbromuron was 0.166 ppm and for all other pesticides was 0.083 ppm.

Liquid chromatography-mass spectrometry (LC-MS) method entitled "Determination of Pesticides in Tea Matrices (Homogenized Leaves and Herbal) using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS) (PMR-011-V1.0)" was recently validated by CFIA laboratories. This method enables the screening of 144 additional pesticide residues which are not detected by GC-MS. The complete list of analytes can be found in Appendix B. The LOD for all measured pesticides was 5.0 ppb except for cyromazine, diclocymet, fuberidazole, ethirmol, methiadathion, quizalofop, thiamethoxam, triforine, and tolyflunanid in which their LOD was 25 ppb. Aclonifen and chlorbromuron had an LOD of 100 ppb.

When used simultaneously the two multi-residue methods can analyse for over 340 different pesticide residues.

#### 2.4.2 Metals Analysis

Using an accredited third party method, all samples were analysed using inductively coupled plasma mass spectrometry (ICP-MS) for the following 18 metals: aluminum, antimony, arsenic, beryllium, boron, cadmium, chromium, copper, iron, mercury, manganese, molybdenum, nickel, lead, selenium, tin, titanium and zinc. Table 2.1 lists the method LOD for the corresponding metal.

To metals.	
Limit of Detection (ppm)	Metal
0.5	Aluminum (Al), Tin (Sn), Zinc (Zn)
0.2	Copper (Cu), Iron (Fe)
0.05	Boron (B), Beryllium (Be), Molybdenum (Mo),
0.05	Manganese (Mn), Antimony (Sb), Titanium (Ti)
0.02	Chromium (Cr), Nickel (Ni), Selenium (Se)
0.005	Arsenic (As), Cadmium (Cd), Lead (Pb)
0.0001	Mercury (Hg)

Table 2.1. Detailed list of LODs used in the ICP-MS method for the determination of18 metals.

#### 2.5 Limitations

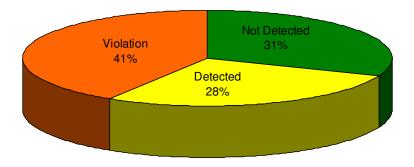
A total of 100 samples were collected and analysed in the 2009-2010 tea pesticide and metals survey. In comparison to the total number of tea products available at the retail level, 100 samples represent a small fraction of products available to consumers regionally. This data is meant to provide a snapshot of the targeted commodities and has the potential to highlight tea commodities that warrant further investigation. Also, this survey does not examine year-to-year trends, impact of product shelf-life or cost of the commodity on the open market. Therefore, no definite conclusions regarding these parameters can be drawn from the data herein.

Analysis was completed on dried tea only. Brewed tea was not tested. Therefore, the results should only be interpreted as dried tea available as sold, and not brewed tea as consumed. The level of transfer of a metal or pesticide from dried tea to brewed tea is dependent on the chemical and physical properties of the compound in question (i.e., solubility). As such, it is difficult to estimate the level of a metal or pesticide that may occur in the brewed tea based on the levels detected in the dried tea.

## **3 Results**

#### 3.1 Pesticide Residue Analysis

A total of 100 tea samples consisting of dried tea leaves and bagged tea were collected at grocery stores and specialty markets. Thirty-one samples did not contain any pesticide residues. Twenty-eight samples contained detected pesticide residues and 41 samples were in violation of the General MRL of 0.1 ppm (Figure 3.1).



#### Figure 3.1. Distribution of dried tea analyses.

In many cases, the 69 tea samples with detected residues contained more than one pesticide residue per sample. The frequency at which specific pesticide residues were found in each sample is illustrated in Figure 3.2. Sixty-two percent of the tea samples contained one and two pesticide residues per sample ( $n^1=28$  and 15, respectively). Thirty-eight percent of the samples contained three to a maximum of seven pesticide residues per sample. The maximum of seven pesticide residues per sample occurred in three samples.

In total there were 166 pesticide residues found on the 69 dried tea samples with detected pesticide residues. There were a number of cases where a single tea sample contained both compliant and non-compliant pesticide residues. Figure 3.2 also shows the number of tea samples in which there was at least one pesticide residue in violation of the 0.1 ppm General MRL. Of the 166 pesticide residues found, 84 (51%) were violations, often with more than one violation per sample. Figure 3.3 shows the number of pesticide residue violations found per sample. Seventy-three percent of the pesticide residue violations were a result of one (n=21) and two (n=9) violations per sample. The remaining 27% of the violations per sample occurred on only one sample. For a detailed list of the samples and pesticide residue violations detected, please refer to Appendix C.

 $<sup>^{1}</sup>$  n = Represents the number of samples

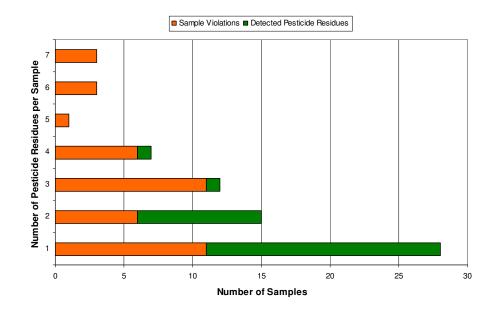


Figure 3.2. Frequency of detected pesticide residues per dried tea sample. Figure shows both the number of samples that contain at least one pesticide residue in violation of established MRLs and samples that contain only detectable pesticide residues.

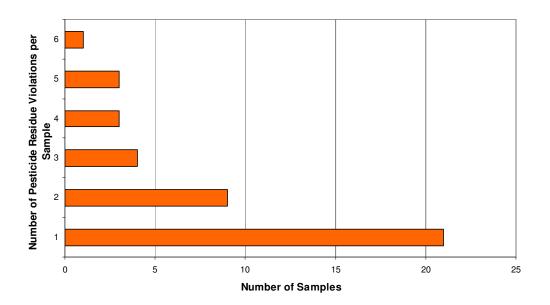


Figure 3.3. Frequency of pesticide residue violations only per dried tea sample.

Since the sampling pattern was not evenly distributed across countries, conclusions regarding the number of pesticide residue violations as a function of country of manufacture cannot be made. However, when the pesticide residue violations were divided by dried tea type, oolong tea contained the highest number of pesticide residue violations at 92% (12 out of 13 samples) (Table 3.1). This was followed by herbal/blends at 58%, green tea at 44%, white tea at 20% and black tea at 8% of those samples analysed. Based on the

samples collected in this targeted survey, oolong tea samples had the highest number of pesticide residue violations per sample at an average of 3.6 violations per sample.

Traditionally, green and black teas are made from tender young tea shoots of one to three leaves and an apical bud; oolong and brick teas are made from tea shoots of three to five leaves and an apical bud<sup>3</sup>. Depending on the pesticide used, the residues can accumulate in the tea leaves resulting in higher levels in mature tea leaves such as those used for oolong tea. The vegetative buds of the green tea leaves may be small at the time of pesticide application, as the leaves grow, the level of pesticide residue per leaf weight is continually reduced until harvest resulting in a lower pesticide residue concentration in green tea<sup>3,4</sup>.

type.		Product Type										
	Number of S	Sample Violatior	ns (Number of P	esticide Residues	s Violations)							
Country of Origin	Black Tea (n=24) <sup>a</sup>	Green Tea (n=41)	Herbal/ Blends (n=12)	Oolong Tea (n=13)	White Tea (n=10)	Total						
Canada <sup>b</sup>	1 (1)	1 (1)				2 (2)						
China	1 (1)	9 (13)	4 (9)	12 (43)	2 (2)	28 (68)						
Hong Kong	—	1 (2)				1 (2)						
Japan	—	3 (3)		_	—	3 (3)						
United Kingdom	—	1 (1)			_	1 (1)						
United States	—	2 (4)	1 (1)		_	3 (5)						
Vietnam	—	1 (1)	2 (2)		—	3 (3)						
Total	2	18 (25)	7 (12)	12 (43)	2 (2)	41 (84)						

Table 3.1. Number of the pesticide residue violations as a result of dried tea sample type.

 $a^{a}$  n = the total number of samples collected.

<sup>b</sup> Tea is not grown in Canada, rather tea leaves are imported for processing and re-sale into domestic and import markets.

In total there were 41 dried tea samples containing 84 pesticide residue violations. The overall compliance rate was 59%. All violations were assessed and appropriate follow-up action was pursued. Exposure to these pesticide residues in tea is not expected to pose a human health concern to consumers given the consumption of tea relative to other food commodities.

#### 3.2 Metals Analysis

The same 100 samples that were analysed for pesticide residues were also analysed for 18 different metals. The 18 metals analysed included aluminum, antimony, arsenic, beryllium, boron, cadmium, chromium, copper, iron, lead, manganese, mercury, molybdenum, nickel, selenium, tin, titanium and zinc. Only those metals of greater interest to human health (i.e., arsenic, chromium, cadmium, lead, mercury) and those for which highest levels were found (i.e., aluminum and manganese) are discussed in the text in detail. The results for all of metal analyses are presented in Appendix D. It should also be noted that the results presented in Appendix D are a measure of the total metal concentration present in the food and do not distinguish between organic and inorganic forms, or ionic species. As such, these results do not provide direct information about the bioavailability or the toxicity of the metal nor do they reveal the potential source (i.e., anthropogenic versus naturally occurring).

Some of the main sources of heavy metals in plants are the soil, fertilizers and pesticides<sup>5</sup>. Please note that the levels of metals reported in this targeted survey are for dried tea only and are comparable to levels found in other academic studies. Transfer rates of 20-80% from dried tea leaves to brewed tea have been found for some of the metals analysed in tea<sup>6</sup>. The level of transfer is often dependent on the chemical and physical properties of the metal in question (i.e., solubility). The CFIA did not undertake this survey to study the levels of these compounds in brewed tea.

#### 3.2.1 Arsenic

The arsenic levels ranged from <LOD to 2.760 ppm in this targeted survey. Dried green tea leaves contained the maximum level of arsenic (2.760 ppm), however oolong tea leaves had the highest average concentration at 0.384 ppm.

In a similar study surveying the arsenic levels in Chinese tea, levels of total arsenic in dried tea leaves varied from below the LOD (0.01 ppm) to 4.81 ppm<sup>7</sup>. In another study arsenic concentrations from 800 different types of Chinese dried tea samples ranged from below the LOD to 4.43 ppm<sup>8</sup>. Even though some of the original tea leaf samples contained high levels of arsenic in these studies, the concentration in the brewed tea was low due to the low leaching ability of arsenic by hot water<sup>7</sup>.

Brewed tea made with samples analysed in this survey would be expected to contain arsenic at levels below those observed in the study cited above. It is noted that these concentrations are below the Canadian Drinking Water Quality Guideline for total arsenic of  $0.010 \text{ mg/L}^9$ .

#### 3.2.2 Cadmium

Cadmium levels in the dried tea leaves from this survey ranged from 0.006 ppm to 0.200 ppm, with dried green tea leaves containing the maximum level at 0.200 ppm. However, oolong tea leaves had the highest average concentration at 0.059 ppm.

In a study investigating 798 Chinese dried tea samples, cadmium levels ranged from <LOD to 1.07 ppm, with the highest average levels observed in green tea<sup>8</sup>. Based on an extensive review article discussing the trace elements in tea leaves (i.e., fresh leaves), made tea (i.e., manufactured dried tea) and tea infusion (i.e., brewed tea), the levels of cadmium found in this targeted survey are consistent with the peer-reviewed literature<sup>8</sup>. In the peer-reviewed literature, it was observed that cadmium levels in brewed tea were generally low<sup>6</sup> and although not directly comparable, it can be noted that these low cadmium levels in tea are within the Canadian Drinking Water Quality Guideline for cadmium of 0.005 mg/L<sup>10</sup>.

#### 3.2.3 Chromium

The chromium levels in the dried tea leaves ranged from 0.058 ppm to 9.423 ppm. The highest level was found in one sample of green tea leaves at 9.423 ppm, while black tea leaves had the highest average chromium levels at 1.394 ppm.

In a study investigating 801 tea samples (i.e., green, black, oolong) collected from the main tea-producing regions in China, chromium levels ranged from below the LOD to 16.1 ppm<sup>6</sup>. In another study investigating the elemental composition of tea leaves and brewed

tea, chromium levels ranged from 3.9 ppm to 6.2 ppm<sup>11</sup>. In this targeted survey the highest average level of chromium was observed in black tea, which is consistent with the latter study. The levels of chromium found in this targeted survey are consistent with the peer-reviewed literature<sup>11, 12</sup>. Different studies assessing the transference of metals into brewed tea observed as much as 42.2% to 67.5% of the measured chromium from the dried leaves leaching into brewed tea<sup>11, 13</sup>.

#### 3.2.4 Lead

In this survey lead was found in 100% of the targeted survey samples with levels ranging from 0.076 ppm to 4.296 ppm, with the maximum in oolong tea leaves. Oolong tea leaves also had the highest average lead concentration at 2.202 ppm.

Studies investigating the heavy metal content of various dried teas from India, China, Turkey and Taiwan reported levels of lead that ranged from 0.04 ppm to 97.9 ppm <sup>5</sup>, <sup>11,13,14,15</sup>. The lead levels found in this survey fall within the range of the peer-reviewed literature. High levels of lead in oolong tea leaves are not unusual, as the older leaves used in the making of oolong tea tend to contain higher concentrations of lead<sup>13</sup>. Studies investigating the transference of lead from dried tea leaves to brewed tea have observed 15.3% to 58.6% release into the brewed tea<sup>11,13</sup>.

#### 3.2.5 Mercury

Mercury was found in 32% of the samples collected with levels ranging from <LOD to 0.030 ppm with the maximum occurring in dried green tea leaves. Dried green tea leaves also exhibited the highest average concentration at 0.004 ppm.

Levels of mercury in tea have not been as widely studied and characterized as other metals. The mercury content in tea leaves of different brands of Indian teas were found to range from 0.004 ppm to 0.032 ppm in a radio-labelled study<sup>16</sup>. The transfer rates after brewing and boiling observed in the radio-labelled study was 19% and 34%, respectively.

#### 3.2.6 Aluminum

The tea leaf is known to accumulate  $aluminum^{6,11}$ . All dried tea samples contained detectable levels of aluminum ranging from 312.9 ppm to 5178 ppm with a mean of 1049 ppm. Oolong dried tea leaves had the highest average aluminum content at 1202 ppm.

The mean levels of aluminum found in the dried tea leaves are comparable to the literature. In a study assessing the metal content in different types of dried teas found a mean aluminum level of 1074 ppm<sup>17</sup>. In Indian dried teas the level of aluminum ranged from ~300 ppm to 6500 ppm13. The same study investigated the rate of transfer at two different times, one minute and five minute infusions. The rate of transfer for the one minute tea infusion was 28.0% and 36.0% for the five minute tea infusion<sup>13</sup>.

#### 3.2.7 Manganese

All dried tea samples collected contained some level of manganese ranging between 184 ppm to 1691 ppm, with an overall average of 886.3 ppm. Oolong tea leaves had the highest average manganese levels at 1128 ppm.

Manganese was found between 148 and 1595 ppm (average 824.8 ppm) in a variety of tea samples from China, Japan, India, Kenya and Sri Lanka<sup>17</sup>. Another study measuring heavy metals in black tea produced in Turkey found manganese levels that ranged from 563.9 to 1081.6 ppm (average 788.0 ppm)<sup>11</sup>. In black teas from India manganese levels ranged from 333 ppm to 406 ppm<sup>13</sup>. This same study also looked at the transfer rates of different elements from the dried tea leaf to brewed tea at two different times, one minute and five minute infusions. The transfer rate for the one minute infusion was 28.4% and 41.6% for the five minute infusion<sup>6</sup>.

## *3.2.8 Beryllium,Copper, Iron, Molybdenum, Nickel, Selenium, Tin, Titanium, Zinc, Boron and Antimony*

The remaining metals that were analysed were beryllium, copper, iron, molybdenum, nickel, selenium, tin, titanium, zinc, boron and antimony. For more detailed results for each individual metal see Table D.

## **4** Conclusions

The 2009-2010 dried tea survey was completed in order to elucidate the levels of pesticide residues and metals in domestic and imported dried tea. In total, 100 black, green, herbal/blends, oolong and white tea samples were purchased at retail. Detectable pesticide residues and quantifiable levels of metals were found in all types of dried tea leaves sampled. In total there were 41 dried tea samples containing at least one pesticide violation. When the pesticide violations were distributed according to dried tea type, oolong tea contained the highest percentage of samples with of pesticide residue violations at 92% followed by herbal blends at 58%, green tea at 44%, white tea at 20% and black tea at 8%. All violations were assessed and appropriate follow-up action was pursued. Exposure to these pesticide residues in tea is not expected to pose a human health concern to consumers given the consumption of tea relative to other food commodities. The overall compliance rate for pesticide residues in dried tea leaves for this targeted survey was 59%.

All 100 samples were analysed for 18 different metals. Beryllium was the only metal that was not detected in any dried tea sample. The levels of metals found in this targeted survey were comparable to other levels found in the related published literature.

### **5** References

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info(%23toc%235040%239999%239999999999999999923FLA%23display%23Articles)&\_cdi=5040&\_s ort=d& docanchor=& ct=84& acct=C000013498& version=1& urlVersion=0& userid=403646&md5=675 dee64ef4ba53b28dde740a5556327&searchtype=a

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**6 Appendix A** Table A1. Combined list of analytes (206) detected by the GC-MS pesticide multi-residue method (PMR-010-V1.0) from the CFIA Calgary Laboratory used in this targeted survey.

		Flowence method	
2,4-DDD (o,p'-DDD)	Cypermethrin	Flamaprop-methyl	Phorate
4',4-DDE	Cyprazine	Flamprop-isopropyl	Phosalone Dhah alimida
a-BHC	Dacthal	Fluchloralin	Phthalimide
a-Chlordane	Deltamethrin	Flumetralin	Pirimicarb
a-Endosulfan	Demeton-O	Fluorochloridone-2	Pirimiphos-ethyl
Alachlor	Demeton-S	Fluorodifen	Pirimiphos-methyl
Aldrin	Demeton-S-methyl	Fonofos	Procymidon
Allidochlor	Des-ethyl Atrazine	Heptachlor	Profenofos
Aramite-1	Desmetryn	Heptachlor Epoxide endo	Profluralin
Aspon	d-HCH	Heptanofos	Promecarb
Atrazine	Di-allate	Hexachlorobenzene	Prometon
B-BHC	Diazinon	Hexazinone	Prometryne
Benalaxyl	Dichlobenil	Imazalil	Pronamide
Bendiocarb	Dichlormid	Iodofenphos	Propachlor
Bendiocarb1	Dichlorvos	Iprodione	Propanil
b-Endosulfan	Diclofluanid	Isazophos	Propargite
Benfluralin	Diclofop-methyl	Isofenphos	Propazine
Benodanil	Dicloran	Isopropalin	Propetamphos
Bifenox (2)	Dicofol	Leptophos	Propham
Bifenthrin	Dicrotophos	Lindane (G-BHC)	Propiconazole
Bromacil	Dicrotophos	Malathion	Prothiophos
Bromophos	Dicrotophos	Metalaxyl	Pyrazophos
Bromophos-ethyl	Dimethachlor	Metazachlor	Quinalphos
Bromopropylate	Dimethoate	Methidathion	Quintozene
Bupirimite	Dinitramine	Methoprotryne	Secbumeton
Butachlor	Dioxathion	Methoxychlor	Simazine
Butralin	Diphenamid	Methyl Trithion	Sulfallate
Butylate	Diphenylamine	Metolachlor	Sulfotep
Carbetamide	Disulfoton	Metribuzin	Sulprophos
Carbofenthion	Edifenphos	Mexacarbate	Tecnazene
Carboxin	Endosulfan Sulphate	Mexacarbate1	Terbacil
Chlorbenzilate	Endrin	Mirex	Terbufos
Chlorbromuron	EPN	Myclobutanil	Terbumeton
Chlorbufam	EPTC	Naled	Terbutryne
Chlorfevinphos	Erbon	Naled-1	Terbutylazine
Chlorflurenol-methyl	Etaconazole-1	Nitrapyrin	Tetrachlorvinphos
Chlormephos	Ethalfluralin	Nitrofen	Tetradifon
Chloroneb	Ethion	Norflurazon	Tetramethrin (2)
Chloropropylate	Ethofumesate	Nuarimol	Thiobencarb
Chlorpropham	Ethoprophos	O,P-DDT	Tolyfluanid
Chlorpyrifos	Ethylan	O-phenylphenol	Trans-Chlordane
Chlorpyrifos-methyl	Etridiazole	Oxadiazon	trans-Mevinphos
Chlorthiamid	Etrimfos	Oxadixyl	trans-Permethrin
Chlorthion	Fenamiphos	Oxychlordane	Triadimefon
Chlozolinate	Fenarimol	Oxyflurofen	Triadimenol
Cis-Mevinphos	Fenchlorophos	p,p'-DDD	Tri-allate
cis-Permethrin	Fenitrothion	P,P'-DDT	Triazophos

(PMR-010-V1.0) from the CFIA Calgary Laboratory used in this targeted survey.						
Clomazone	Fenpropathrin	Parathion	Tribufos			
Crufomate	Fenson	Pebulate	Trifluralin			
Cyanazine	Fensulfothion	Penconazole	Vernolate			

Pendimethalin

Phenthoate

Vinclozolin

Table A1. Combined list of analytes (206) detected by the GC-MS pesticide multi-residue method

Cycloate Note:

Cyanophos

Pesticide residues highlighted in bold are detected in both the GC-MS and LC-MS analytical methods.

Fenthion

Fenvalerate

## 7 Appendix B

#### Table B1. List of analytes (160) detected by the LC-MS pesticide multi-residue method (PMR-011-V1.0) at the CFIA Calgary laboratory.

3-Hydroxy-carbofuran	Diethofencarb	Isocarbamide	Pyridalyl
Abamectin B1 <sup>a</sup>	Difenoconazole	Isoprocarb	Pyridaphenthion
Acetochlor	Dimethametryn <sup>f</sup>	Isoxathion	Pyridate
Actonifen	Dimethomorph	Linuron	Pyrifenox
Aldicarb	Diniconazole		Pyrimethanil
Aldicarb sulfone	Dioxacarb	Mepanipyrim Mephosfolan	Pyriproxyfen
Aldicarb sulfoxide		Methabenzthiazuron	Quinoxyfen
	Dipropetryn		
Azaconazole	Diuron	Methidathion	Quizalofop
Benomyl <sup>a, b</sup>	Dodemorph	Methiocarb	Quizalofop-ethyl <sup>f</sup>
Benoxacor	Emamectin <sup>e</sup>	Methiocarb sulfone	Schradan f
Bitertanol	Epoxiconazole	Methiocarb sulfoxide	Spinosyn A <sup>d</sup>
Bromuconazole	Ethiofencarb	Methomyl	Spinosyn D <sup>d</sup>
Butafenacil	Ethiofencarb sulfone <sup>f</sup>	Methoxyfenozide	Spirodiclofen
Butocarboxim sulfoxide	Ethiofencarb sulfoxide	Metolcarb	Spiromesifen
Cadusafos	Ethirimol	Metoxuron	Spiroxamine <sup>e</sup>
Carbaryl	Ethoprop	Mexacarbate	Sulfentrazone
Carbendazim	Etofenprox	Molinate	Tebufenozide <sup>f</sup>
Carbendazim d3	Etoxazole	Monocrotophos	Tebufenpyrad
Carbendazim d4	Fenamidone	Napropamide	Tebupirimfos
Carbofuran	Fenazaquin	Naptalam	Tepraloxydim
Carbofuran d3	Fenhexamid	Neburon	Tetraconazole
Carbosulfan <sup>c</sup>	Fenoxanil	Ofurace	Thiabendazole
Carfentrazone-ethyl	Fenpropidin <sup>e</sup>	Oxadixyl	Thiabendazole d4
Chlorbromuron	Fenpropimorph	Oxamyl	Thiacloprid
Chloridazon	Fenpyroximate	Oxamyl-oxime	Thiamethoxam
Chlorimuron-ethyl	Fentrazamide	Oxycarboxine	Thiazopyr
Chloroxuron	Fluazifop-butyl	Paclobutrazol	Thiodicarb
Chlorthiamid	Flucarbazone Sodium	Pencycuron	Thiofanox
Chlortoluron	Flutolanil	Penoxsulam	Thiofanox sulfone
Clodinafop-propargyl	Flutriafol	Picolinafen	Thiofanox sulfoxide
Cloquintocet-mexyl	Forchlorfenuron	Picoxystrobin	Thiophanate-methyl <sup>a,b</sup>
Clothianidin	Formetanate	Piperophos	Tolylfluanid
Cyanofenphos	Fosthiazate	Pretilachlor	Tralkoxydim
Cycloxydim	Fuberidazole	Primisulfuron-methyl	Trichlorfon
Cycluron	Furathiocarb	Prodiamine	Tricyclazole
Cyromazin	Haloxyfop <sup>f</sup>	Propamocarb	Trietazine
Demeton-S-methyl sulfone	Imazamethabenz-methyl	Propoxur	Trifloxysulfuron
Demeton-S-methyl sulfoxide	Imidacloprid <sup>f</sup>	Pymetrozine	Triforine
Desmedipham	Indoxacarb	Pyraclostrobin	Trimethacarb
Diclocymet	Iprovalicarb	Pyraflufen-ethyl	Zinophos

Notes: Pesticide residues highlighted in bold are detected in both the GC-MS and LC-MS analytical methods.

<sup>a</sup> Prepare fresh standard solutions for benomyl, carbosulfan, formetanate and thiophanate methyl.

<sup>b</sup> Any detected benomyl and/or thiophanate-methyl will be reported as carbendazim.
<sup>c</sup> Any detected carbosulfan will be reported as carbofuran.

<sup>d</sup> Spinosad is a mixture of spinosyn Å and spinosyn D; quantitation can be based on either one.

<sup>e</sup> Retention time may change over time.

<sup>f</sup> For Tea Leaves (only) these pesticides showed interference peaks in the matrix blanks, not quantifiable.

## 8 Appendix C

Table C1. Detailed list of pesticide violations found in dried tea samples	Table C1. Detailed list of	pesticide violations	found in dried	tea samples.
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Table C1. Detailed list of pesticide violations found in dried tea samples.     Particide Desides     Sample Number     Amount (num)									
Pesticide Residue	Sample Number	Amount (ppm)	Sample Number	Amount (ppm)					
	2009TEA0011A	0.16	2009TEA0047A	1.10					
	2009TEA0016A	0.32	2009TEA0051A	1.00					
	2009TEA0017A	0.27	2009TEA0061A	0.41					
Bifenthrin	2009TEA0023A	0.28	2009TEA0070A	0.19					
	2009TEA0031A	0.21	2009TEA0076A	2.00					
	2009TEA0036A	0.25	2009TEA0079A 2009TEA0080A	0.24					
	2009TEA0042A	1.10	2009 I EA0080A	0.11					
	2009TEA0046A	0.22		0.66					
Carbendazim	2009TEA0051A 2009TEA0061A	0.22	2009TEA0083A 2009TEA0094A	0.00					
	2009TEA0016A	0.23	2009TEA0074A	0.46					
Cypermethrin	2009TEA0047A	0.70	2009TEA0076A	0.73					
Cypermetiinii	2009TEA0051A 2009TEA0061A	0.72	2009TEA0079A 2009TEA0090A	0.12					
	2009TEA0061A 2009TEA0070A	0.30	2009 I EA0090A	0.12					
		0.70	2000TE & 0051 &	0.70					
Endosulfan Total	2009TEA0046A	0.70	2009TEA0051A	0.70					
	2009TEA0047A		2009TEA0061A	0.12					
Etofenprox	2009TEA0052A	0.26		1.10					
Fenpropathrin	2009TEA0068A	0.13	2009TEA0074A	1.10					
	2009TEA0011A	0.24	2009TEA0080A	0.20					
Fenvalerate	2009TEA0065A	0.13	2009TEA0086A	0.17					
	2009TEA0066A	0.18	2009TEA0090A	0.15					
	2009TEA0074A	0.62							
	2009TEA0012A	0.10	2009TEA0065A	0.25					
	2009TEA0015A	0.19	2009TEA0070A	0.14					
	2009TEA0016A	0.11	2009TEA0074A	0.45					
	2009TEA0023A	0.18	2009TEA0076A	0.88					
Ter. 1. 1	2009TEA0035A	0.10	2009TEA0077A	0.20					
Imidacloprid	2009TEA0037A	0.12	2009TEA0079A	0.54					
	2009TEA0043A	0.24	2009TEA0080A	0.19					
	2009TEA0046A	0.83	2009TEA0082A	0.23					
	2009TEA0047A	<u> </u>	2009TEA0083A	0.26					
	2009TEA0053A		2009TEA0089A	0.30					
	2009TEA0061A	0.95	2009TEA0091A						
	2009TEA0017A	0.16	2009TEA0061A	0.44					
Methomyl	2009TEA0036A	0.13	2009TEA0076A	0.18					
	2009TEA0046A	0.93	2009TEA0091A	0.12					
	2009TEA0047A	1.08	2000TE 4 0050 4	0.24					
o,p'-DDT	2009TEA0074A	0.12	2009TEA0079A	0.24					
Propargite	2009TEA0022A	0.15	1						
D i	2009TEA0067A	1.40	2009TEA0091A	0.44					
Propazine	2009TEA0084A	0.28	2009TEA0100A	0.52					
	2009TEA0089A	0.31	· · · · · · · · · · · · · · · · · · ·						
Thiacloprid	2009TEA0012A	0.17	2009TEA0045A	0.18					
r	2009TEA0033A	0.28	2009TEA0057A	0.44					
Triazophos	2009TEA0079A	0.18							

Note: Those sample number highlighted in bold have multiple pesticide residue violations.

## 9 Appendix D

Metal Analyte	Total #	#	# Not	Min	Max	Mean
and Tea Type	Samples	Detected	Detected	(ppm)	(ppm)	(ppm)
Aluminum	100	100	0	312.9	5178	1049
Black Tea	24	24	0	502.4	1974	1026
Green Tea	41	41	0	312.9	5178	1111
Herbal/Blends	12	12	0	399.1	1501	709.6
Oolong Tea	13	13	0	817.0	1565	1202
White Tea	10	10	0	431.0	1791	1062
Antimony	100	60	40	<lod< td=""><td>0.227</td><td>0.030</td></lod<>	0.227	0.030
Black Tea	24	9	15	<lod< td=""><td>0.111</td><td>0.019</td></lod<>	0.111	0.019
Green Tea	41	27	14	<lod< td=""><td>0.227</td><td>0.040</td></lod<>	0.227	0.040
Herbal/Blends	12	10	2	<lod< td=""><td>0.076</td><td>0.031</td></lod<>	0.076	0.031
Oolong Tea	13	9	4	<lod< td=""><td>0.066</td><td>0.028</td></lod<>	0.066	0.028
White Tea	10	5	5	<lod< td=""><td>0.055</td><td>0.019</td></lod<>	0.055	0.019
Arsenic	100	99	1	<lod< td=""><td>2.760</td><td>0.191</td></lod<>	2.760	0.191
Black Tea	24	24	0	0.025	0.727	0.111
Green Tea	41	40	1	<lod< td=""><td>2.760</td><td>0.213</td></lod<>	2.760	0.213
Herbal/Blends	12	40	0	0.051	0.277	0.213
Oolong Tea	12	12	0	0.051	0.277	0.120
White Tea	10	10	0	0.000	0.050	0.384
			100			
Beryllium Black Tea	<b>100</b> 24	<b>0</b> 0	24	<lod <lod< td=""><td><lod <lod< td=""><td><lod <lod< td=""></lod<></lod </td></lod<></lod </td></lod<></lod 	<lod <lod< td=""><td><lod <lod< td=""></lod<></lod </td></lod<></lod 	<lod <lod< td=""></lod<></lod 
Green Tea	24 41	0	24 41	<lod <lod< td=""><td><lod <lod< td=""><td><lod <lod< td=""></lod<></lod </td></lod<></lod </td></lod<></lod 	<lod <lod< td=""><td><lod <lod< td=""></lod<></lod </td></lod<></lod 	<lod <lod< td=""></lod<></lod 
Herbal/Blends	41	0	41 12	<lod <lod< td=""><td><lod <lod< td=""><td><lod <lod< td=""></lod<></lod </td></lod<></lod </td></lod<></lod 	<lod <lod< td=""><td><lod <lod< td=""></lod<></lod </td></lod<></lod 	<lod <lod< td=""></lod<></lod 
	12					
Oolong Tea		0	13	<lod< td=""><td><lod< td=""><td><lod< td=""></lod<></td></lod<></td></lod<>	<lod< td=""><td><lod< td=""></lod<></td></lod<>	<lod< td=""></lod<>
White Tea	10	0	10	<lod< td=""><td><lod< td=""><td><lod< td=""></lod<></td></lod<></td></lod<>	<lod< td=""><td><lod< td=""></lod<></td></lod<>	<lod< td=""></lod<>
Boron	100	100	0	<b>7.610</b>	27.14	13.23
Black Tea Green Tea	24 41	24 41	0 0	11.10 8.728	27.14 18.97	15.30 13.21
Herbal/Blends	41 12	41 12	0		16.13	13.21
	12	12	0	9.346 7.610	10.13	12.03
Oolong Tea White Tea	13 10	13 10	0	8.863	12.74 14.89	10.93
Cadmium Black Tea	<b>100</b> 24	<b>100</b> 24	<b>0</b> 0	<b>0.006</b> 0.014	<b>0.200</b> 0.118	<b>0.050</b> 0.034
Green Tea	24 41	24 41	0	0.004	0.118	0.054
					0.200	
Herbal/Blends Oolong Tea	12 13	12 13	0 0	0.009	0.003	0.043 0.059
White Tea	13 10	13	0	0.031 0.017	0.090	0.059
Chromium						
Black Tea	<b>100</b> 24	<b>100</b> 24	<b>0</b> 0	<b>0.058</b> 0.235	<b>9.423</b> 3.953	<b>0.737</b> 1.394
Green Tea	24 41	24 41	0		5.935 9.423	
Herbal/Blends	41 12	41 12	0	$0.058 \\ 0.154$		0.713 0.266
Oolong Tea	12	12	0		$0.667 \\ 0.744$	0.200
White Tea	13 10	13	0	0.108 0.129	1.221	0.289
Copper Black Tea	<b>100</b> 24	<b>100</b> 24	<b>0</b> 0	<b>5.116</b> 8.552	<b>26.61</b> 25.77	<b>12.67</b> 14.19
Green Tea	24 41	24 41	0	8.332 5.340	26.61	14.19
Herbal/Blends	41 12	41 12	0	3.340 10.39	20.01 17.97	12.77
Oolong Tea	12	12	0	5.116	17.97	7.834
-	13 10	13 10	0	8.230	13.26	12.28
W/ hite 1 49		117	U	0.230	10.00	12.20
White Tea				AE EL	1015	100 1
Iron Black Tea	<b>100</b> 24	<b>100</b> 24	<b>0</b> 0	<b>45.76</b> 84.29	<b>1317</b> 270.1	<b>199.1</b> 149.6

Metal Analyte and Tea Type	Total # Samples	# Detected	# Not Detected	Min (ppm)	Max (ppm)	Mean (ppm)
Green Tea	<b>3 3 amples</b> 41	41	0	45.76	846.5	225.4
Herbal/Blends	41 12	41 12	0	43.76 91.29	840.3 477.7	160.8
Oolong Tea	12	12	0	91.29 84.17	477.7	309.4
White Tea	13 10	13	0	84.17 74.79	190.5	112.7
Lead <sup>c</sup>	100	100	0	0.076	4.296	1.201
Black Tea	24	24	0	0.127	2.899	0.637
Green Tea	41	41	0	0.076	3.747	1.305
Herbal/Blends	12	12	0	0.292	2.475	1.138
Oolong Tea	13	13	0	1.016	4.296	2.202
White Tea	10	10	0	0.195	1.643	0.902
Manganese	100	100	0	184.0	1691	886.3
Black Tea	24	24	0	184.0	1411	787.9
Green Tea	41	41	0	244.2	1691	888.9
Herbal/Blends	12	12	0	408.1	1017	691.9
Oolong Tea	13	13	0	782.6	1489	1128
White Tea	10	10	0	470.7	1636	1031
Mercury	100	32	68	<lod< td=""><td>0.030</td><td>0.003</td></lod<>	0.030	0.003
Black Tea	24	4	20	<lod< td=""><td>0.015</td><td>0.002</td></lod<>	0.015	0.002
Green Tea	41	17	24	<lod< td=""><td>0.030</td><td>0.004</td></lod<>	0.030	0.004
Herbal/Blends	12	5	7	<lod< td=""><td>0.010</td><td>0.003</td></lod<>	0.010	0.003
Oolong Tea	13	3	10	<lod< td=""><td>0.007</td><td>0.001</td></lod<>	0.007	0.001
White Tea	10	3	7	<lod< td=""><td>0.006</td><td>0.002</td></lod<>	0.006	0.002
Molybdenum	100	87	13	<lod< td=""><td>2.128</td><td>0.070</td></lod<>	2.128	0.070
Black Tea	24	22	13	<lod< td=""><td>0.229</td><td>0.076</td></lod<>	0.229	0.076
Green Tea	41	34	7	<lod< td=""><td>0.229</td><td>0.040</td></lod<>	0.229	0.040
Herbal/Blends	41 12	12	0	0.022	2.128	0.033
Oolong Tea	12	12	3	<lod< td=""><td>0.090</td><td>0.038</td></lod<>	0.090	0.038
White Tea	10	9	1	<lod <lod< td=""><td>0.090</td><td>0.038</td></lod<></lod 	0.090	0.038
Nickel	100	100	0	1.280	8.808	3.692
Black Tea	24	24	0	1.280	6.966	4.207
Green Tea	41	41	0	1.336	8.808	4.038
Herbal/Blends	12	12	0	1.896	4.249	3.364
Oolong Tea	13	13	0	1.461	5.740	2.352
White Tea	10	10	0	1.706	4.446	3.175
Selenium	100	97	3	<lod< td=""><td>0.432</td><td>0.096</td></lod<>	0.432	0.096
Black Tea	24	24	0	0.032	0.124	0.072
Green Tea	41	38	3	<lod< td=""><td>0.209</td><td>0.079</td></lod<>	0.209	0.079
Herbal/Blends	12	12	0	0.038	0.123	0.072
Oolong Tea	13	13	0	0.055	0.432	0.232
White Tea	10	10	0	0.029	0.107	0.077
Tin	100	56	44	<lod< td=""><td>0.522</td><td>0.114</td></lod<>	0.522	0.114
Black Tea	24	14	10	<lod< td=""><td>0.522</td><td>0.150</td></lod<>	0.522	0.150
Green Tea	41	17	24	<lod< td=""><td>0.268</td><td>0.066</td></lod<>	0.268	0.066
Herbal/Blends	12	9	3	<lod< td=""><td>0.478</td><td>0.212</td></lod<>	0.478	0.212
Oolong Tea	12	12	1	<lod< td=""><td>0.170</td><td>0.154</td></lod<>	0.170	0.154
White Tea	10	4	6	<lod< td=""><td>0.228</td><td>0.057</td></lod<>	0.228	0.057
Titanium	100		1	<lod< td=""><td></td><td>2.087</td></lod<>		2.087
Black Tea	100 24	<b>99</b> 24	1 0	<b><lod< b=""> 0.947</lod<></b>	<b>13.04</b>	
					6.973	2.467
Green Tea	41	40	1	<lod< td=""><td>13.04</td><td>2.302</td></lod<>	13.04	2.302
Herbal/Blends	12	12	0	0.777	3.704	1.577
Oolong Tea	13	13	0	0.548	3.397	1.519

Metal Analyte and Tea Type	Total # Samples	# Detected	# Not Detected	Min (ppm)	Max (ppm)	Mean (ppm)
White Tea	10	10	0	0.868	5.026	1.642
Zinc	100	100	0	14.48	56.63	27.30
Black Tea	24	24	0	18.01	31.77	24.30
Green Tea	41	41	0	14.48	56.63	28.56
Herbal/Blends	12	12	0	23.29	39.45	31.84
Oolong Tea	13	13	0	14.95	42.53	24.67
White Tea	10	10	0	20.38	36.60	27.28