

## SPEX CertiPrep App Note

### Examination of Commercial Botanical Products for the Presence of Heavy Metals by ICP-MS

#### Abstract

The consumption of botanical products has increased over the past two decades as consumers trend to what are perceived to be natural and high quality botanical products. The primary regions of spice and tea production around the world have often been cited as having less stringent safety and quality standards in regards to consumer products. Products from these regions have been noted to contain a variety of adulterants and contaminants including wear metals and toxic elements.

Common botanicals (black & red pepper, cinnamon, mustard, cumin, and turmeric) sold as spices, teas, condiments, and supplements were purchased at dollar stores, farmers markets, chain stores, and online vitamin outlets. Products selected covered the range of preparations including ground species, blends, supplement teas, and sauces (retail and 'organic') products. Physical and chemical screening methods were used to detect gross adulteration and counterfeiting. ICP was used to determine the macroelement components (Si, Na, Mg, Fe, and K) that indicated possible adulteration or contamination. High levels of bulking agents, including silica and sodium, were often found in low cost spice and botanical samples indicating potential adulteration. ICP-MS was used to determine the presence and level of heavy metal contamination and adulteration. Most of the spice groups studied had many examples of high heavy metals content at the ppm level including very high lead levels which could be indicative of adulteration by lead chromate or lead oxides.

#### Methods & Materials

##### Samples

- 7 Spice Groups & Products (Supplements, Teas, Sauces, and Condiments)
  - Black Pepper
  - Red Peppers: Spice, Hot Sauce, Chili Powder
  - Cinnamon: Spice, Supplement, Tea
  - Ginger: Spice, Supplement, Tea
  - Cumin: Spice, Curry Powder
  - Mustard Seed: Spice, Condiment
  - Turmeric: Spice, Supplement
- Whole and Ground Spices
- Range of Prices
  - Dollar Store, Farmers Market, Grocery, Retail Chain, Name Brand, Organic
- SPEX CertiPrep Standards:
  - CLMS-1: Multi-Element Solution Standard 1
  - CLMS-2: Multi-Element Solution Standard 2
  - CLMS-3: Multi-Element Solution Standard 3
  - CLMS-4: Multi-Element Solution Standard 4
- Reagents:
  - High Purity Nitric Acid
  - Hydrofluoric Acid

# Examination of Commercial Botanical Products for the Presence of Heavy Metals by ICP-MS

## Solid Sample Preparation

- Sample Grinding: SPEX SamplePrep 6970 EFM Freezer/Mill
  - General Program
    - 2.5 g of plastic
    - Pieces cut into < 5 mm
  - Program
    - Precool for 20 minutes
  - Grind for 5 cycles (2 minutes per cycle)
  - Each cycle = 2 minute cooling
  - Impact Rate: 16 impacts/second
- Sample Digestion: CEM Mars 5 Microwave with Easy Prep Vessels
  - 0.1 g sample with 10 mL HNO<sub>3</sub>
  - 15 minute ramp to 210 °C
  - 15 minute hold
  - Some incomplete digestions of PVC & sample residue filtered and weight subtracted from samples
- Instrumentation:
  - Perkin Elmer ICP-OES - Wear Metals
  - Agilent ICP-MS 7700
    - Cyclonic spray chamber
    - Analysis performed
    - Normal mode: air
    - Collision mode: helium

## Results & Conclusion

The most traded spice in the world is black pepper and accounts for 20% of the world spice market. Only Cr (total) and Pb were found to any significant level in the black pepper samples.

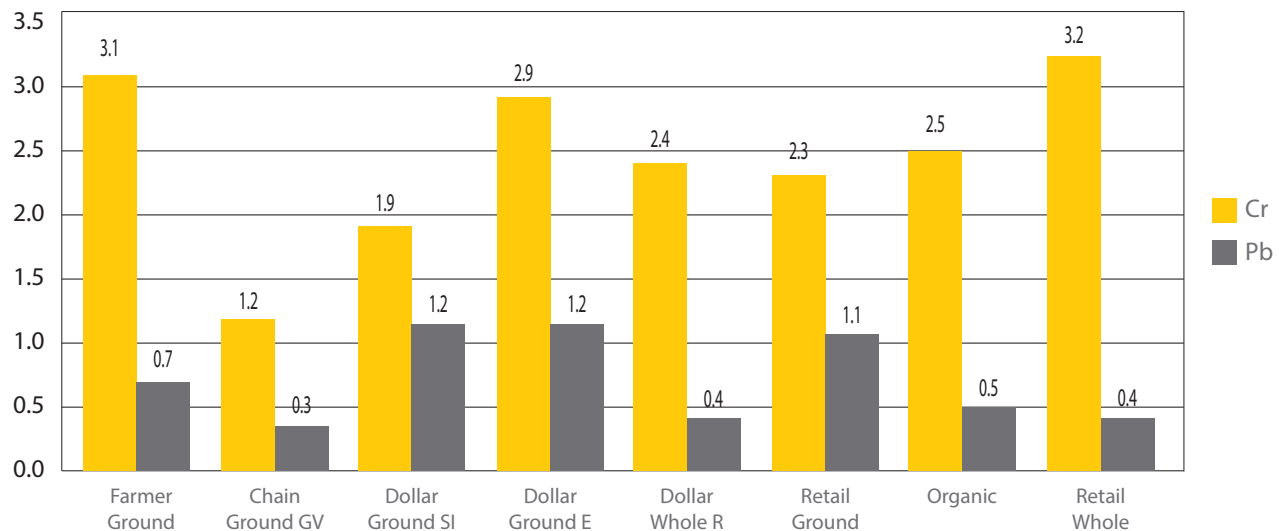


Figure 1. Chromium (total) and Lead Levels in Brands of Black Pepper (µg/g).

Red pepper is also a highly and widely used spice in the world. The spectrum of red pepper and red pepper products ranges from ubiquitous “Ground Chili Peppers” and “Red Pepper Flakes” to “Smoked Paprika”, “Cayenne Peppers” and all the worlds hot pepper sauces and blends.

The examination of heavy metals in the red pepper spices shows a trend where the more highly processed the spice, the more heavy metal content was found. Red pepper flakes, which are generally minimally processed or ground before packaging, had the lowest levels of heavy metal including possible wear metal constituents such as Cr (total) and Pb. The more highly ground and processed red peppers (i.e. chili, cayenne and paprika) had much higher heavy metal content especially for Cr (12 µg/g) and Pb (> 2 µg/g) for dollar store paprika (Figure 2).

# Examination of Commercial Botanical Products for the Presence of Heavy Metals by ICP-MS

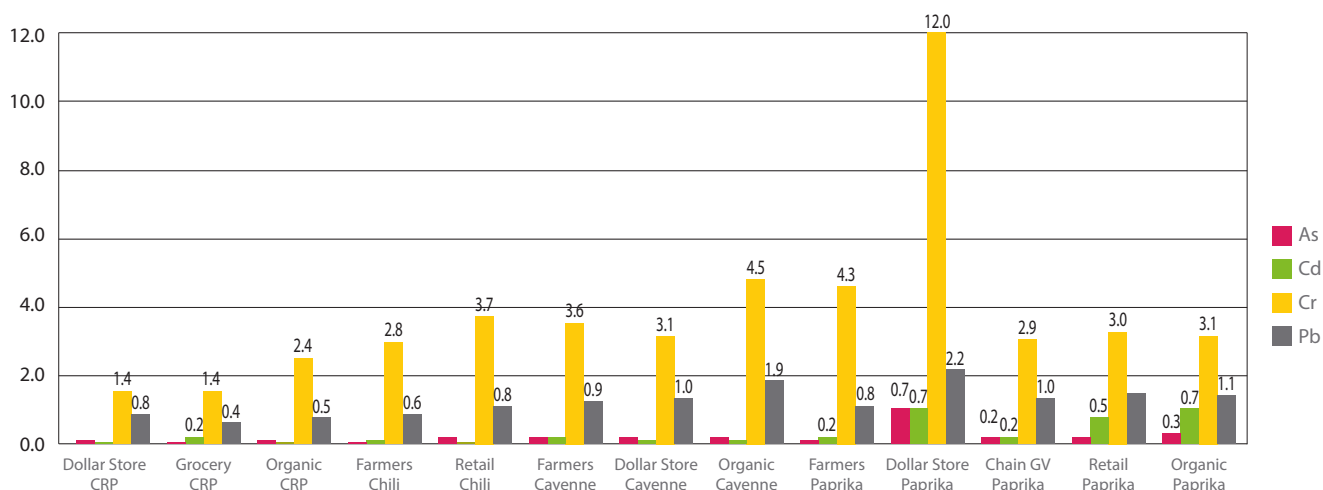


Figure 2. Heavy Metals in Various Forms of Red Pepper Spices: Crushed Red Pepper (CRP), Chili Peppers, Cayenne Peppers and Paprika (µg/g).

The heavy metal content of the red pepper showed some small amounts of As and Cd, but not Hg. The most abundant heavy metals were Cr (total) and Pb. Retail brands had overall lowest amounts of heavy metals. The Chinese food hot sauce packet, the chain store hot sauce and the fast food hot sauce packet showed the highest amounts of Pb (from 0.7-1.0 µg/g) (Figure 3).

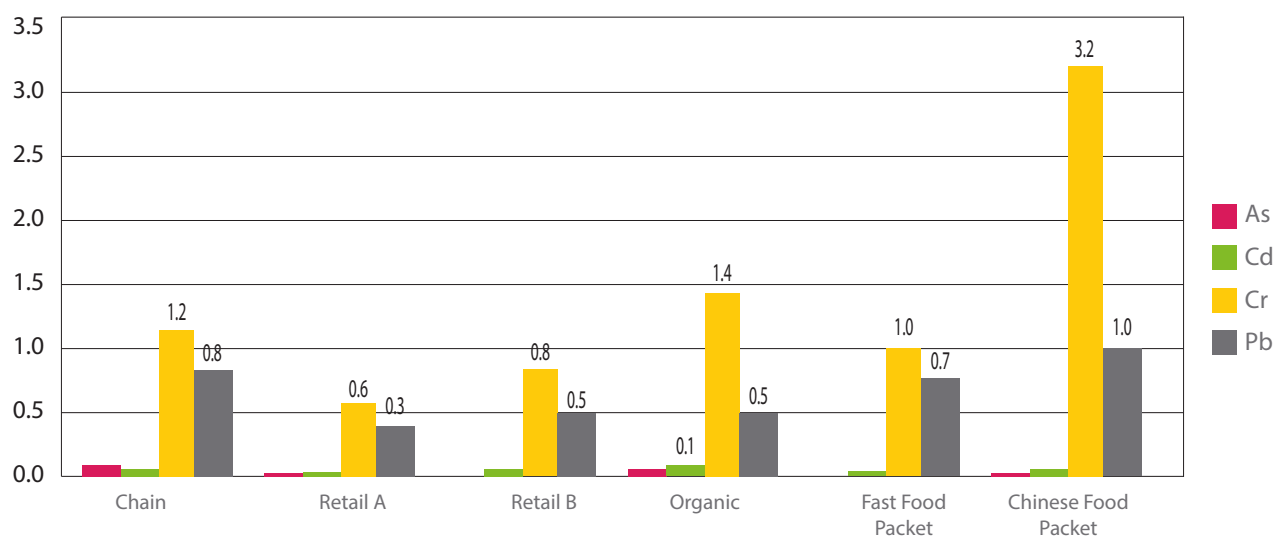


Figure 3. Heavy Metal Content in Chain, Retail, Organic, Fast Food, and Chinese Food brands (µg/g)

The result of the potentially high amounts of lead in red pepper products are a dramatically increased exposure to lead when consumed in normal use. The average size of a fast food packet of condiment ranges from about 10 g to 20 g which roughly corresponds to a generous tablespoon of either red pepper flakes or ground spice. An intake of about 20 g of spice or sauce exposes the user to between 15 g to 21 g of Pb from sourced such as the Chinese food packet, fast food packet and chain hot sauce.

Overall the exposure of Pb and Cr is highest for the hot sauces out of the red pepper spices, red pepper spice blends and hot sauces. A 20 g dose from two packets of the Chinese food hot sauce would expose a 150 lb adult to over 25% of the Allowable Daily Limit (ADL) for lead (Figure 4).

# Examination of Commercial Botanical Products for the Presence of Heavy Metals by ICP-MS

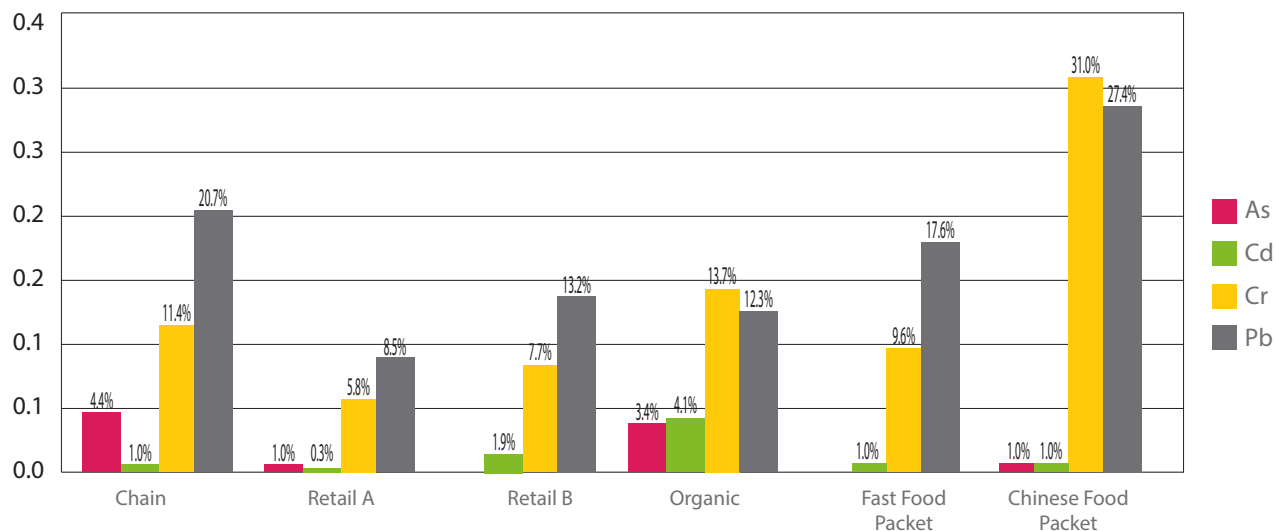


Figure 4. Allowable Daily Limit (% ADL) for Heavy Metal Exposure from Hot Sauce ( 20 g serving).

The heavy metals, As, Cd, Cr, Hg, and Pb, were found in all of the spice samples. Hg was only found in small quantities in a few samples of mustard seed, mustard condiment, cinnamon, and ginger. The most prominent heavy metals across all samples were Cr (total) and Pb. Lead was most prevalent in the cinnamon and turmeric samples (2.7-2.8  $\mu\text{g/g}$ ). Cadmium was the highest in the chili powder blends at almost double the amount found in all of the other products (1.2  $\mu\text{g/g}$ ). Arsenic was found at up to 0.7  $\mu\text{g/g}$  in the red pepper spices (paprika) (Figure 5).

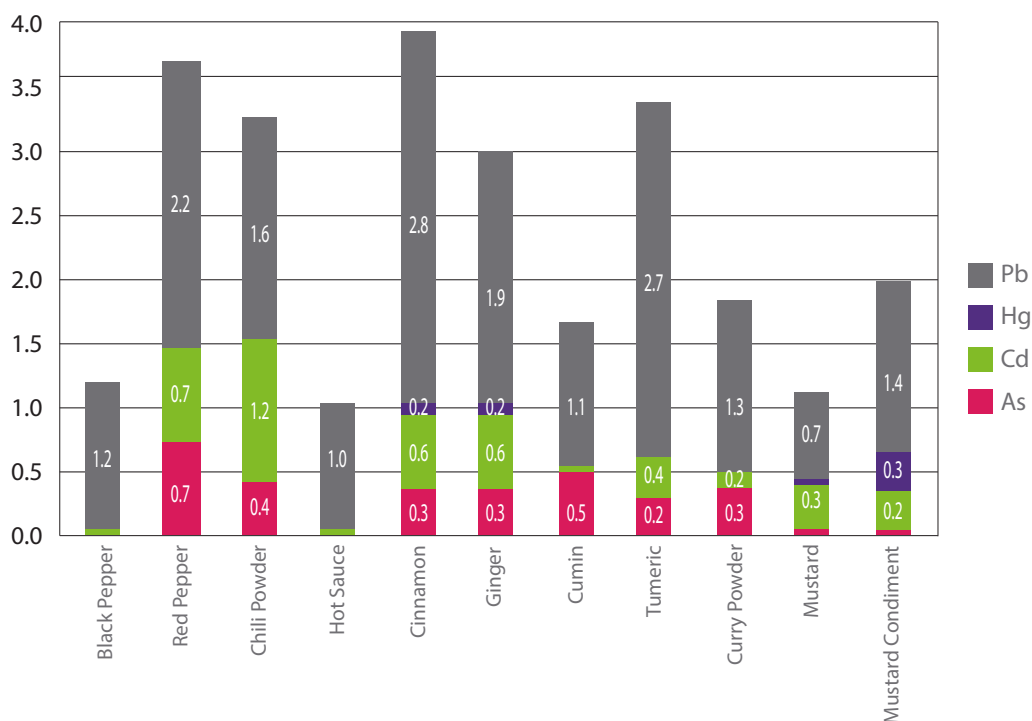


Figure 5. Comparison of Heavy Metals in All Spices and Spice Products Studied ( $\mu\text{g/g}$ ).

# Examination of Commercial Botanical Products for the Presence of Heavy Metals by ICP-MS

The distribution of heavy metals and wear metals within the spice groups showed that, overall, the highest heavy metal and wear metal concentrations were found in the discount and less expensive brands. Almost 70% of the highest heavy metal content was found in the dollar store and budget spice products. This trend can indicate an intentional adulteration of spices for economic gain as opposed to just random environmental exposure.

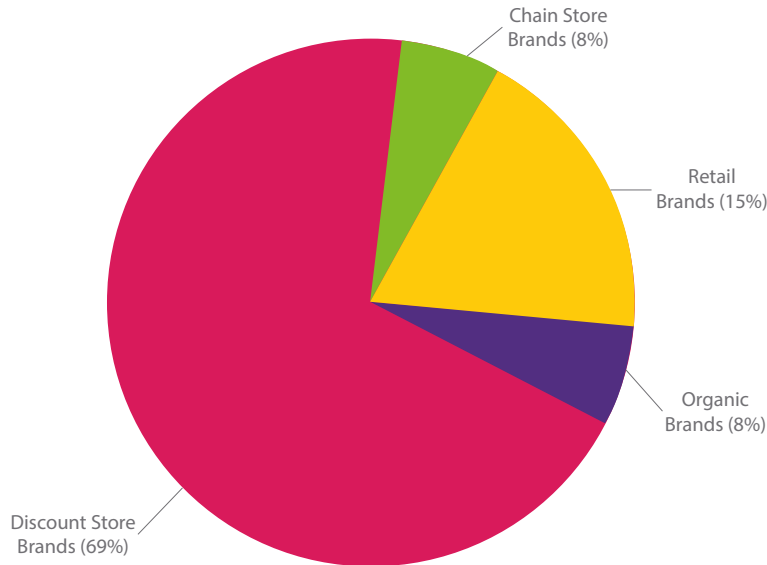


Figure 6. Heavy Metal Content Distribution Among Brand Types.

Heavy metals, in particular, lead and total chromium, were found in significant concentrations in the majority of the spice groups tested. Spices such as cinnamon and turmeric were found to have the highest concentration of heavy metals such as lead. But the potential exposure from minute amounts of spices used in most products is relatively small when compared to the exposure from some of the spice products such as the blends and condiments. The highest exposure to lead was found in the hot sauce condiments which are customarily used in doses above 10 g to 20 g. The free hot sauce packets given out at Chinese food restaurants contributed over 25% of an adults Allowable Daily Limit (ADL) for lead. Other hot sauces, including a fast food hot sauce and a chain store bottled hot sauce, were also of concern for the potential lead exposure.

The trend of the heavy metal and wear metal concentrations showed a pattern where less expensive or bargain brands were at more risk for higher heavy metal or wear metal levels suggesting a potential for intentional adulteration or lack of proper quality control.

## Contact Us

Phone: 800.LAB.SPEX • 732.549.7144 • Fax: 732.603.9647  
CRMSales@spex.com • spexcertiprep.com

© 2020 SPEX CertiPrep. All Rights Reserved.

CONNECT WITH US

