SPEX CertiPrep App Note

Analysis of Cannabis and Hemp Products for Heavy Metals

Introduction

The cannabis industry has taken the world by storm and has flooded the market with new products. Recently, concerns have arisen around the safety of this largely unregulated market. Cannabis testing laboratories emerged to fill the need for specialized testing for cannabinoid potency, pesticides, bacteria/mold and other potential contaminants. Sadly, a significant group of contaminants has been largely ignored: toxic metals.

Recreational cannabis and hemp are both the same species. Federally legal hemp products are easy to obtain by the general public. Hemp products are also used as base for cannabis products & cannabinoid extracts. However, due to a ban on hemp cultivation in the US, virtually all of the hemp used in the US is imported from China, India, Eastern Europe, and Canada. Studies of other commodities exported from these countries have reported widespread heavy metal contamination (i.e. spices, teas, grains, etc.).

Cannabis plants are potential bioaccumulators of heavy metals. In the production of these products, a large amount of plant material is processed to extract concentrates and oils, thereby increasing the risk of heavy metal contamination. The scope of this study was to analyze various legal hemp products, currently on the market, for heavy metal contamination and use hemp as a model for methods development for restricted products. Samples were digested using microwave digestion and analyzed by ICP-OES and ICP-MS.

Methods and Materials

Samples:

Hemp oils were purchased online from various sources including vitamin distribution companies, CBD suppliers, and online auction sites. Two of the samples were encapsulated hemp oil and two samples were hemp product extracts in another matrix such as olive oil or alcohol base. One sample was identified as an essential oil (see Table 1).

Table 1. Hemp Oil Sample Sources and Descriptions.

ID	Form	Purchased From	Description	Source
1	Supplement Oil	Vitamin Distribution Company	Cold Pressed Unrefined Hemp Seed Oil	Canada
2	Supplement Oil	Vitamin Distribution Company	Cold Pressed Unrefined Hemp Seed Oil	Canada
3	Supplement Oil	Vitamin Distribution Company	Cold Pressed Unrefined Hemp Seed Oil - Organic	Canada
4C	Capsule	Vitamin Distribution Company	Cold Pressed Unrefined Hemp Seed Oil	Canada
5	Supplement Oil	Vitamin Distribution Company	Cold Pressed Virgin Hemp See Oil - Organically Grown	No Info
6	Supplement Oil	Vitamin Distribution Company	Artisan Cold Pressed - Organic	No Info/Made in USA
7 & 19	Supplement Oil	Vitamin Distribution Company	Cold Pressed - Organic	Canada
8E (CBD)	Extract Supplement	CDB Cannabinoid Producer	Premium Hemp Extract Supplement (5000) in Olive Oil	Colorado
9E (CBD)	Extract Supplement	CDB Cannabinoid Producer	Hemp Classic Concentrate (1500 mg) Tincture	Colorado
10	Supplement Oil	Auction Site	100% Pure Virgin Organic Hemp Seed Oil	No Info
11C	Capsule	Vitamin Distribution Company	Hemp Oil from Premium Hem Seeds, Cold Pressed	Canada
12	Supplement Oil	Auction Site	Organic Virgin Hemp Seed Oil, Cold Pressed Unrefined	Canada
13	Supplement Oil	Auction Site	Unrefined Hemp Seed Oil, Cold Pressed - Organic	No Info
14	Supplement Oil	Auction Site	Organic Virgin Hemp Seed Oil	No Info
15	Supplement Oil	Auction Site	Organic Hemp Seed Oil	No Info
16	Supplement Oil	Auction Site	Organic Virgin, Non-GMO Hemp Seed Oil	No Info
17	Essential Oil	Hemp Online Supplier	Cannabis Essential Oil 100% Clean, Chemical Free Sun Grown	No Info
18	Industrial	Hemp Online Supplier	Industrial Hemp Seed Oil	No Info

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Sample Preparation

Sample Digestion:

- Samples were digested using a CEM Mars 5 Microwave
  - Microwave Conditions
    - Easy Prep Vessels & XP Vessels
    - 0.2 g sample
    - 10 mL HNO_3
      - Some samples 1-2 drops HF
      - 15 minute ramp to 210 °C
      - 15 minutes hold

Materials:

- SPEX CertiPrep Standards
  - CLMS-1, CLMS-2, CLMS-3, CLMS-4: Multi-Element Solution Standards 1-4
  - USP <232> Standards: USP-TXM2, USP-TXM3, USP-TXM5
- Reagents
  - High Purity Nitric Acid

Instrumentation:

- Perkin Elmer ICP-OES - Macroelements
- Agilent ICP-MS 7700 - Heavy Metals
  - Meinhard Nebulizer
  - Cyclonic Spray Chamber
  - Analysis Performed
    - Normal Mode: Air
    - Collision Mode: Helium

Method Design:

This study was designed to examine heavy metal contamination present in edible hemp oil products used in nutraceutical preparations. The samples were first examined by ICP for macroelement content and to determine possible ICP-MS interferences for heavy metal quantitation. Limits for heavy metals imposed by various organizations (FDA, EPA, AHPA, USP) were used as benchmarks for heavy metal exposure. Most limits were designed to apply to a 150 lb (68 kg) adult (Table 2). The AHPA (American Herbal Products Association) has produced some of the most stringent limits we found, specifically for cannabis products.

Table 2. Common Heavy Metal Exposure Limits for 150 lb (68 kg) Adults (µg/day).

Source	EPA	EPA	FDA	AHPA (2012)	ATSDR	USP <232>	Min	Max
Route	Oral RfD	Drinking Water	Bottled Water	Oral	Oral	Oral		
Daily Limit	70 kg - Adult	2 (L)	2 (L)	70 kg - Adult	70 kg - Adult	Daily		
As	21	20	20	10	21	15	10	21
Cd	70	10	10	4.1	14	5	4.1	70
Pb	None	30	10	6	10	5	5	30
Hg	7	4	4	2	21	30	2	30

** Red denotes speciation limits*

In some cases, hemp and cannabinoid products have been popularized for the treatment of children with seizure disorders and other childhood illnesses. The limits for heavy metals have not been generally examined for their application to children's health. For the purpose of understanding the possible exposure limits for children, the adult levels were calculated to the weight of a 23 kg child (approximately 50 lb) (Table 3).

Table 3. Calculated Heavy Metal Exposure Limits for Children (23 kg) (µg/day).

Source	EPA	AHPA (2012)	ATSDR	USP <232>	Min	Max
Route	Oral RfD	Oral	Oral	Oral		
Daily Limit	23 kg - Child (Calculated)	23 kg - Child (Calculated)	23 kg - Child (Calculated)	Daily		
As	7	3.3	7	15	3.3	15
Cd	23	1.4	5	5	1.4	23
Pb	None	2	3.33	5	2	5
Hg	2.3	0.66	7	30	0.66	30

** Red denotes speciation limits*

Results & Discussion

Heavy Metal Concentrations in Hemp Samples

The highest concentration of any of the heavy metals was chromium at over 1,800 ppb of total chromium. The chromium species were not separated or calculated as separate species. The overall, most prevalent heavy metal detected in the hemp samples was lead with samples ranging from 13 to 137 ppb. Arsenic was also found in all of the samples tested from 19 to 137 ppb. Cadmium levels ranged from below detection limits up to 25 ppb. Mercury was only found in one sample at 16 ppb (Table 4).

Table 4. Heavy Metal Concentrations Found in Hemp Products (µg/kg).

ID	As	Cd	Cr (total)	Hg	Pb	Description
1	19	*	32	*	69	Oil
2	25	7	68	*	50	Oil
3	22	5	43	*	47	Oil
4C	23	10	46	*	64	Capsule
5	27	13	163	*	33	Oil
6	32	15	68	*	67	Oil
7	39	15	157	*	137	Oil
8E	42	7	53	*	65	Extract
9E	137	5	43	*	13	Concentrate
10	25	10	102	*	89	Oil
11C	20	25	50	*	90	Capsule
12	25	7	63	*	36	Oil
13	24	13	59	*	50	Oil
14	28	*	1832	*	96	Oil
15	*	26	*	*	21	Oil
16	21	24	38	*	65	Oil
17	31	6	39	16	43	Oil
18	29	*	30	*	15	Industrial Oil
Mean	**33**	**12**	**170**	**16**	**58**	-
Min	**19**	*	**30**	*	**13**	-
Max	**137**	**26**	**1832**	**16**	**137**	-

† Red denotes highest result detected of each element.
** Below detection level*

Heavy Metal Concentration and Dosage

The suggested dosage for the various products was on average 1-2 tsp up to twice daily for oils, which calculated to about a minimum of 28 grams per day dosage. The dosage suggested on the extracts was one or more 0.5 mL doses, twice a day. This dosage was calculated out to be about a minimum of 1 gram per day dosage. The capsule dosage called for up to four 1,000 mg capsules a day (4 g dosage).

The concentrations measured in the hemp oil were calculated to give a final concentration of heavy metals for the stated dosages. Arsenic concentrations, which ranged from 19 to 137 ppb, gave final exposures of 5 to 11% of an adult's daily exposure limit. However, for children, that exposure increased to between 16 to 32% of the calculated daily limit for arsenic. Cadmium, which previously had concentrations up to 25 ppb, provided up to 16% of an adult's daily cadmium limit, and up to 48% of a child's potential daily limit (Table 5).

Table 5. Comparison of Arsenic and Cadmium Exposure to Adult and Calculated Child Daily Limits.

#	As (µg/dose)	% Adult Daily Limit (As)	% Child Daily Limit (As)	Cd (µg/dose)	% Adult Daily Limit (Cd)	% Child Daily Limit (Cd)
1	0.53	5	16	DL	-	-
2	0.69	7	21	0.20	5	15
3	0.60	6	18	0.15	4	11
5	0.65	6	19	DL	-	-
6	0.89	9	27	0.41	10	30
7	1.08	11	32	0.41	10	30
10	0.71	7	21	0.27	7	20
12	0.70	7	21	0.21	5	15
13	0.67	7	20	0.36	9	26
14	0.79	8	24	DL	-	-
15	DL	-	-	0.74	-	-
16	0.58	6	17	0.66	16	48
17	0.71	7	21	0.17	4	12
18	0.82	8	25	DL	-	-
19	1.01	10	30	DL	-	-
Adult Limit	**10.00**	**-**	**-**	**4.10**	**-**	**-**
Child Limit	**3.33**	**-**	**-**	**1.37**	**-**	**-**

The lead levels found in the oils were of the most concern when compared to daily limits. For an adult, there is a 5 µg limit per day. Several samples of nutritional or medicinal hemp oil were found to give 50% more of an adults limit. For children, many of the oil samples were well over a potential daily limit for a child. A 28 g dose of Oil #7 would have provided over 200% of a potential daily limit for a child (Figure 1).

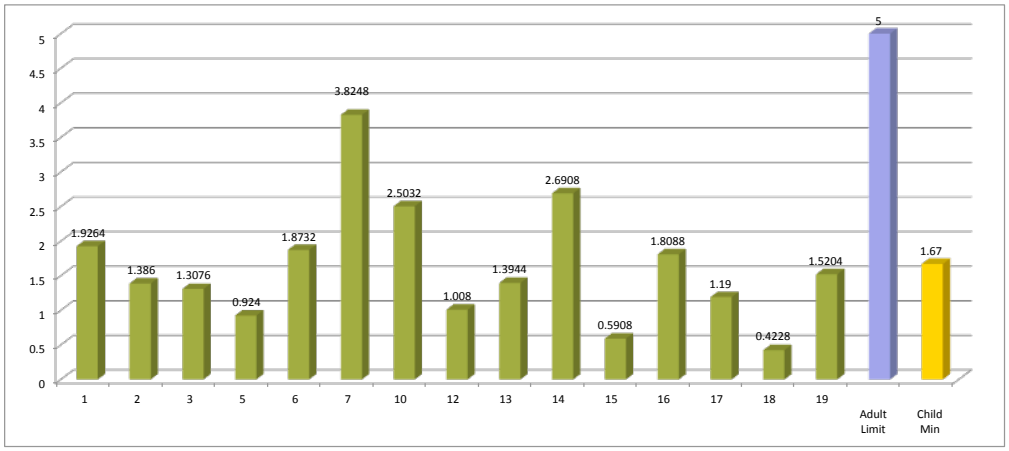

Figure 1. Lead Concentration Found in Hemp Oils (µg/dose).

The extracts and capsules did also have some heavy metal concentrations, but for both the adult and child limits the concentrations were well under potentially problematic limits (Figure 2).

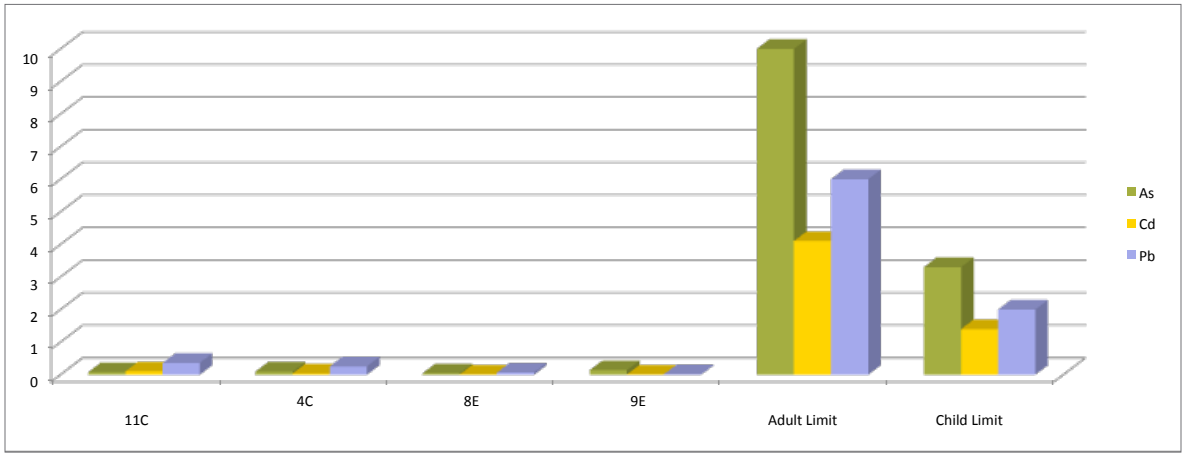

Figure 2. Heavy Metal Exposure in Daily Doses of Hemp Product Extracts and Capsules (µg/dose).

Conclusion

Each of the hemp products studied had some concentration of heavy metals. The lead was the most prevalent heavy metal found in the products. The concern for these heavy metal concentrations comes from the dosage of these common nutraceutical products. The dosages suggested are fairly large and instructions are absent on dosage for children. Possibly, there is a misconception that the product is safe at all dosages due to its pretense of being a supplement or natural product. The limits imposed by organizations such as the APHA, who are attempting to create limits for cannabis products, do not necessarily provide limits which are applicable for a child's exposure to heavy metals through cannabis or hemp products. By using adult limits and calculating them against the body weight of a child, the exposure to heavy metals from these products can be potentially very high. Products that at first did not exceed the heavy metal limits for adults could then be seen as potentially hazardous to a child, especially a child with health concerns.

Contact Us

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