

## APPLICATION NOTE

### Trace Metal Analysis of Commercial Pet Food for Toxic Metals by ICP and ICP-MS

With kind permission of Ralph Obenauf, Patricia Atkins, Lazlo Ernyei & William Driscoll of SPEX CertiPrep, Inc.

The pet food recalls of 2007 increased awareness of the inadequate testing of ingredients in the pet food industry. Pet food is a multi-billion dollar a year business divided into four or five large corporations which produce 80% of US pet food and smaller premium or gourmet pet food companies which produce the remaining 20%.

The quality of many of the ingredients used for pet food is often considered to be inferior or unfit for human consumption. "Premium" brands claim to have superior ingredients and quality. Claims of the quality of premium ingredients do not offer data as to the potential toxicity of elements which may be found in those ingredients. The purpose of our study was to examine pet foods from various sources to determine if they contained potentially toxic elements and if higher quality ingredients equated to less toxic elements present in the food.

This study is not a comprehensive study of all potential contaminants found in pet foods. The random samples tested were deemed to be snapshots of the overall levels of toxic elements that could be consumed by pets.

#### Materials Used

- Standards SPEX CertiPrep standards: CLMS-2N: Multi Element Solution Standard, CL-ICV-1: Initial Calibration Verification Standard.
- 59 pet food samples were donated by the employees from SPEX CertiPrep & SPEX SamplePrep. These samples were purchased from local NJ supermarkets, chain stores, budget stores and pet supply stores.
- 3 human canned proteins: sardines, tuna & chicken
- Brands ranged from discount store brands to 'premium' or 'gourmet' brands
- Prices range: \$0.02 cents/oz to \$0.42 cents/oz
- Foods represented both wet and dry foods for cats and dogs
  - 31 Dry food samples: 18 Dry Dog food samples and 13 Dry Cat food samples,
  - 27 Wet food samples: 13 Wet Dog food samples and 14 Wet Cat food samples

#### Sample Preparation

12 to 16 g of dry pet food samples were ground to an uniform powder consistency using SPEX SamplePrep Freezer/Mill under cryogenic conditions. 6875 Freezer/Mill, 6885 Mid-Size Poly-Vial with the 6888 Mid-Size Vial Adapter. The same operating program was used for all samples: Precool: 10 minutes, Cycles: 5, Grinding time: 2 minutes, Cooling time: 1 minutes, Rate: 12 Cycles per second (CPS).

#### Wet Food Sample Preparation

Wet pet food was homogenized in a blender then blended to smooth consistency. If the food was unable to be completely homogenized, DI water was added 10 mL at a time until smooth consistency was achieved. Weight of water added was later subtracted from sample weight. Samples were tested and calculated as served and not by dry weight basis.

#### Sample Digestion and Analysis

Samples digested in CEM Mars 5 Microwave Unit XP-1500 Vessels

**Microwave program:** Ramp to 200°C over 20 minutes. Pressure maximum: 600 psi Power 100% to 1600 Watts. Hold for 15 minutes. Digestion blanks run on each vessel prior to sample digestion. Digest of samples were diluted 250 to 1000 times before analysis on ICP-MS.

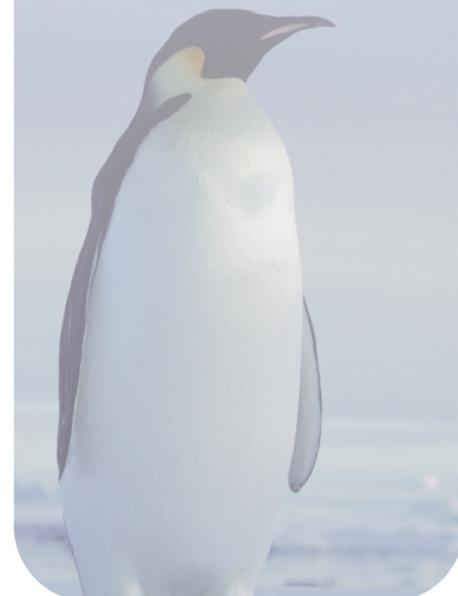
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APPARATUS:  
**Freezer/Mill®**

APPLICATION: Food Safety



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## Instrument Conditions (See Table 1.)

Macroelements measured using Perkin Elmer ICP-OES Optima 7300

Samples run for trace elements on two different ICP-MS:

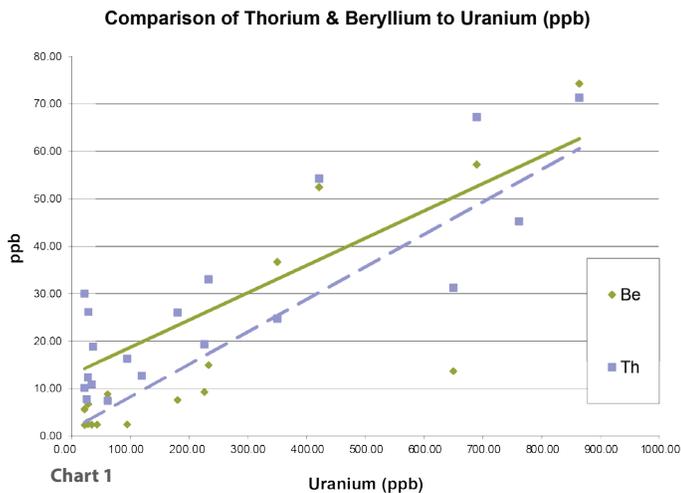
- Perkin Elmer ICP- MS Elan 6000 using Meinhard nebulizer with cyclonic spray chamber.
- Perkin Elmer ICP-MS DRC Elan 6100 using Meinhard nebulizer with cyclonic spray chamber.

## Results: Toxic Trace Elements (See Table 2)

- Large variability of composition between samples
- High ppb / low ppm levels of 11 out of 17 elements examined found in the samples.
- >1 ppm: Cr, Mo, Ni, Pb, Se, Sn & V
- 0.5 ppm to 1 ppm: Co, Hg, Sb, & U
- Human food compared favorably to pet food in terms of toxic metal contamination.
- Toxic elements in human food samples were at levels generally less than those in pet food.

## Results: Uranium (See Chart 1)

- Uranium levels above 250 ppb were found in six samples.
- All the samples with >250 ppb of U were dry dog foods.
- Four of the six samples contained over 500 ppb of U with two samples containing just over 1 ppm of Uranium.
- Samples with high uranium also contained some of the highest levels of Beryllium and Thorium detected in the samples.



## Conclusions: EPA (RfD) Reference Dosage Comparison

### Toxic Element Exposure for Cats

A 10-lb cat eating 1 cup a day (100 g) of dry food or 1 small can of wet food (175 g) with the maximum contamination would be consuming about:

- 29 µg Arsenic (>20x RfD limit)
- 13 µg Cadmium (>3x RfD limit)
- 5 µg Mercury (>10x RfD limit)
- 2 µg Thallium (>3x RfD limit)
- 42 µg Uranium (>3x RfD limit)

Dry cat food contained more contamination which exceeded human RfD guidelines than wet cat food.

Condition	ICP-MS DRC 6100 Perkin Elmer	ICP-MS 6000 Perkin Elmer
Power	1150 W	1000 W
Plasma Gas	15 L/min	15 L/min
Aux Gas	1.2 L/min	1 L/min
Nebulizer Gas	0.89 L/min	0.92 L/min
Sampling rate	1.8 mL/min	1 mL/min
As-1 (DRC)	75.0	-
Be	9.0	9.0
Cd	111, 112, 113, 114	111, 112, 113, 114
Co	-	59.0
Cr (DRC)	52, 53	52, 53
Cs	-	133.0
Cu	-	63, 65
Fe	-	54, 55, 56
Hg	199, 200, 201, 202	199, 200, 201, 202
Mo	-	92, 94, 96, 97, 98, 100
Ni	-	60, 61, 62
Pb	206, 207, 208	206, 207, 208
Sb	121, 123	-
Se	76, 77, 82	76, 77, 82
Sn	116, 117, 118, 119, 120	-
Th	232.0	-
Tl	203, 205	203, 205
U	238.0	238.0
V (DRC)	51.0	51.0

\* ICP-OES used for macro element composition determination

Table 1

Element	Pet Food Min (ppb)	Pet Food Mean (ppb)	Pet Food Max (ppb)	Human Tuna (ppb)	Human Sardines (ppb)	Human chicken
As	4.4	95	290	14	30	4.4
Be	2.0	8.6	74	6.1	3.7	2.9
Cd	1.8	42	130	36	14	1.8
Co	23	200	920	23	44	25
Cr	15	480	2500	25	41	20
Cs	1.1	9.0	28	14	16	2.7
Hg	ND	6.22	54.6	52.5	9.15	ND
Mo	6.2	550	2300	6.2	9.3	23
Ni	48	980	3200	180	380	950
Pb	3.2	210	5900	7.2	11	3.2
Sb	0.92	75	970	0.90	1.6	1.2
Se	64	330	1500	360	320	147
Sn	5.8	350	9400	98	28	5.8
Th	0.06	14	87	ND	0.10	0.08
Tl	1.0	4.0	10	1.0	3.1	1.8
U	0.19	91	860	0.20	6.0	0.20
V	5.2	280	7400	6.2	5.2	5.6

Table 2

## Conclusions: Toxic Element Exposure for Dogs

A 50-lb dog eating 5 cups (500 g) a day of dry food or 1 large can of wet food (375 g) with the maximum contamination would be consuming about:

- Co: 7 samples between 0.7 and 1 ppm
- Cr: 9 samples  $\geq$  1 ppm, highest = 2.5 ppm
- Mo: 12 samples  $\geq$  1ppm, highest = 2 ppm
- Ni: 27 samples  $\geq$  1ppm, highest = 3 ppm
- Pb: 2 samples  $\geq$  1ppm, highest = 6 ppm
- Sb: 1 sample @ 1ppm
- Sn: 6 samples  $\geq$  0.5 ppm, highest = 9 ppm
- V: 4 samples  $\geq$  1ppm, highest = 2 ppm

## Trace Elements found in Pet Foods

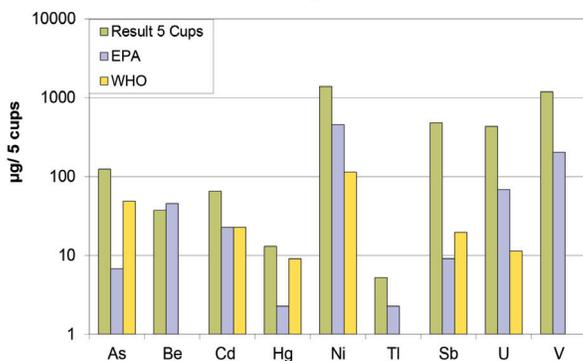
- Concentrations of toxic trace metals were highest in dry food vs. wet food as served.
- Dry dog food had the largest number of significant toxic metals overall.
- Wet cat food had the overall lowest number of toxic metals.
- Seven samples of pet food contained significant amounts of Uranium from 500 to 1000 ppb.
- Significantly high concentrations of toxic metals were found in many of the food samples:

Highest results for trace toxic metals were:

- 9 ppm of Sn in one dry cat food
- 6 ppm of Pb in another dry cat food

## Daily intake from Dry Dog Food

Comparison of Daily Intake of Metals in 5 cups of Dry Dog food to EPA & WHO Human Daily Intake Limits (PTDI)

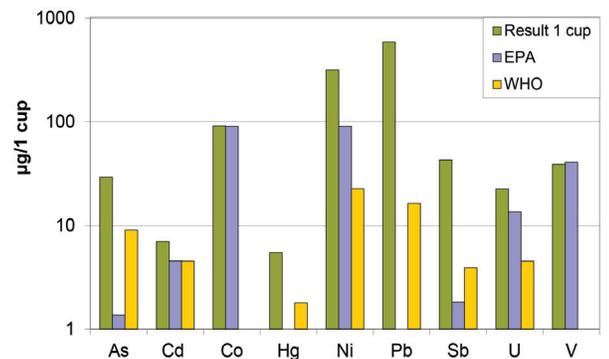


Element	µg in one cup of dry cat food (100g)	EPA RfD per 10 lbs body weight	% EPA	WHO Human PTDI per 10 lbs body weight	% WHO
As	124	6.8	1819	48.6	255
Be	37	45.5	82	-	-
Cd	65.1	22.7	286	22.7	286
Hg	13	2.3	565	9.08	143
Ni	1388	454.5	305	113.6	.
Tl	5.2	2.3	231	-	-
Sb	482.3	9.1	5306	19.5	2468
U	432	68.2	634	11.4	5575
V	1188.77	204.5	581	-	-

Results are for total Cr, EPA RfD is for Cr VI

## Daily intake from Dry Cat Food

Comparison of Daily Intake of Metals in 1 cup of Dry Cat food to EPA & WHO Human Daily Intake Limits (PTDI)



Element	µg in one cup of dry cat food (100g)	EPA RfD per 10 lbs body weight	% EPA	WHO Human PTDI per 10 lbs body weight	% WHO
As	29	1.4	2149	9.1	323
Cd	7	4.5	154	4.5	154
Co	91.6	90.9	101	-	-
Hg	5.5	0.5	1100	1.8	305
Ni	319	90.9	351	22.7	1404
Tl	0.5	0.45	119	-	-
Pb	591.0	-	-	16.3	3616
Sb	43.0	1.8	2361	3.9	1099
U	23	13.6	166	4.5	498
V	39	40.9	95	-	-

Results are for total Cr, EPA RfD is for Cr VI



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