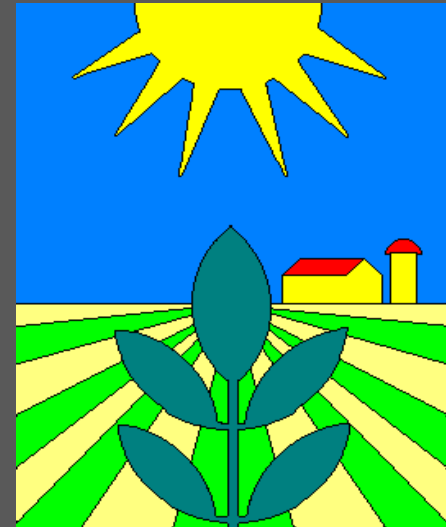


Multi-element Method for Analyzing Animal Feeds and Pet Foods with Microwave Digestion and ICP-MS

New York State Department of Agriculture &
Markets Food Laboratory



Project: A method for the determination of total minerals in animal feed, feed ingredients, forage grain and pet food.

- A need exists for a method that includes additional analytes and potentially additional technologies.
- Elements of low priority should be included if possible; however when a compromise in the multi-analyte method is necessary it should favor the high priority elements.

Elements Analyzed

Original Method

- Al, As, Cd, Cr, Co, Hg, Mo, Ni, Se, V, Sb, U, Be, Th and Tl.

New Method

- Al, As, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Mo, Ni, P, K, Se, Na, S, V, Zn, Sb, U, Be, Th and Tl.

Method

○ Extraction

- Weigh 0.25g of sample in microwave vessel
- Add 5mLs HNO_3 , 2mL H_2O_2 , 1 mL DDI
- Rest for 15 minutes, then vortex
- Digest samples in closed vessel microwave
- Transfer sample to centrifuge tube
- Centrifuge for 10 minutes
- Mix 1mL supernatant with 9mLs DDI

○ Analysis

- Samples are analyzed by ICP-MS

Thermo iCap Q

- He KED (Kinetic Energy Discrimination) reduces interference
- No need to run hydrogen mode
- Standard mode for Be – lower mass
- Reduced rinse time
- Auto tune

Study Materials

SRM 1567b Wheat Flour

SRM 1573a Tomato Leaves

14c2576 Feed Scratch Grain

1432 Poultry Layer

1430 Medicated Swine Starter

1428 Dry Cat Food

1422 Medicated Swine Grower

1321 Dry Dog Food

1426 Medicated Pelleted Sheep Concentrate

1429 Equine Feed

Table 1. Recommended Method Performance Characteristics:

High Priority Elements	Target Concentration, mg/kg		Accuracy, %		Repeatability, % (CV _r)		Reproducibility, % (CV _R)	
	LOQ	Operational Range	at 2x LOQ	at midrange	at 2x LOQ	at midrange	at 2x LOQ	at midrange
Aluminum,	20	20 - 2000	80 - 115	90 - 108	< 9	< 6	< 18	< 12
Arsenic	0.2	0.2 - 60	70 - 125	80 - 115	< 18	< 10	< 36	< 20
Boron,	15	15 - 300	80 - 115	85 - 110	< 9	< 8	< 18	< 16
Cadmium	0.05	0.05 - 20	75 - 120	80 - 115	< 22	< 12	< 44	< 24
Calcium	50	50 - 30000	85 - 110	92 - 105	< 8	< 4	< 16	< 8
Chromium	0.02	0.02 - 60000	70 - 125	92 - 105	< 25	< 4	< 50	< 8
Cobalt	0.01	0.01 - 50	70 - 125	80 - 115	< 30	< 10	< 60	< 20
Copper	0.4	0.4 - 1000	75 - 120	90 - 108	< 16	< 7	< 32	< 14
Fluorine	0.03	0.03 - 300	70 - 125	85 - 110	< 25	< 8	< 50	< 16
Iodine	0.01	0.01 - 800	70 - 125	85 - 110	< 30	< 7	< 60	< 14
Iron	5	5 - 6000	80 - 115	90 - 108	< 12	< 6	< 24	< 12
Lead	0.5	0.5 - 200	75 - 120	85 - 110	< 16	< 8	< 32	< 16
Magnesium	60	60 - 16000	85 - 110	92 - 105	< 8	< 4	< 16	< 8
Manganese	1	1 - 4000	75 - 120	90 - 108	< 15	< 6	< 30	< 12
Mercury	0.01	0.01 - 4	70 - 125	75 - 120	< 30	< 15	< 60	< 30
Molybdenum	0.02	0.02 - 300	70 - 125	85 - 110	< 25	< 8	< 50	< 16
Nickel	0.01	0.01 - 500	70 - 125	85 - 110	< 30	< 8	< 60	< 16
Phosphorus	160	160 - 20000	85 - 110	92 - 105	< 7	< 4	< 14	< 8
Potassium	150	150 - 40000	85 - 110	92 - 105	< 7	< 4	< 14	< 8
Selenium	0.01	0.01 - 10	70 - 125	80 - 115	< 30	< 13	< 60	< 26
Sodium, (NaCl)	100 Na	100 - 120000	85 - 110	95 - 102	< 8	< 3	< 16	< 6
Chloride (NaCl)	300 Cl	300 - 120000	90 - 108	95 - 102	< 7	< 3	< 14	< 6
Sulfur	50	50 - 10000	85 - 110	92 - 105	< 8	< 5	< 16	< 10
Vanadium	0.005	0.005 - 100	70 - 125	85 - 110	< 32	< 9	< 64	< 18
Zinc	0.3	0.3 - 2000	75 - 120	90 - 108	< 18	< 6	< 36	< 12

The limits of quantitation are based on the lowest Maximum Tolerable Level (MTL) as published in the 2005 NRC Mineral Tolerances in Animal Health for various species. For optimal quantitation, each LOQ was designated as 1/10 of the lowest MTL.

Element	MTL mg/kg	Animal Studied	LOQ mg/kg	Range mg/kg
Thallium	0.65	Rat	0.065	0.065-30
Beryllium	1.0	Dog	0.1	0.1-1000
Thorium	10	Mice	1.0	1-8000

2* LOQ

		certified	mean	mean	Preferred	n = 3	Preferred
<u>sample #</u>	<u>element</u>	<u>value ppm</u>	<u>result ppm</u>	<u>% Rec</u>	<u>%Rec</u>	<u>CVr</u>	<u>CVr</u>
1567b	Ca	191.4	202.53	105.82	85-110	11.83	<8
1567b	Cu	2.03	2.03	100.00	75-120	8.14	<16
1567b	Fe	14.11	13.47	95.44	80-115	8.80	<12
1567b	Mg	398	401.67	100.92	85-110	14.41	<7
1567b	Mn	9	9.19	102.07	80-115	9.62	<12
1567b	P	1333	1390.00	104.28	90-108	12.10	<6
1567b	K	1325	1340.00	101.13	90-108	10.99	<6
1567b	S	1645	1501.33	91.27	90-108	11.18	<6
1567b	Zn	11.61	11.87	102.21	80-115	11.22	<12
1573a	Na	136	119.33	87.75	85-110	6.35	<8
1573a	Ca	50500	52833.00	104.62	92-105	1.33	<3

Midrange

<u>sample #</u>	<u>element</u>	<u>spiked conc ppm</u>	<u>mean % Rec</u>	<u>Preferred %Rec</u>	<u>n = 3 CVr</u>	<u>Preferred CVr</u>
1432b	Mn	~2000	114.41	90-108	5.85	<6
1432b	Zn	~1000	119.35	90-108	6.22	<6
2576c	Al	~1010	108.18	90-108	2.42	<6
2576c	Be	~500.1	108.84	90-108	11.96	<7
2576c	Tl	~15	106.95	80-115	1.96	<12
2576e	Hg	~2	85.46	75-120	5.78	<16
2576f	Sb	~153	111.11	85-110	2.68	<8
2576f	Cd	~10	113.41	80-115	11.21	<12
2576f	Se	~5	119.82	75-120	18.55	<13
2576f	Cr	~500	108.08	85-110	10.39	<7
2576g	Mo	~150	106.29	85-110	2.00	<8
2576g	Ni	~250	105.67	85-110	2.25	<8
2576h	V	~50	103.51	80-115	2.10	<9
2576h	U	~105	106.42	85-110	3.67	<8
2576h	As	~30	108.08	80-115	4.19	<12
2576i	Co	~25	110.20	80-115	4.60	<12
1432a	Cu	~500	98.58	85-110	5.53	<7
1432a	Fe (n=2)	~3000	81.46	90-108	5.89	<6
1432c	B	~157	111.47	85-110	6.54	<8
1430	Mg	~4000	124.22	90-108	5.42	<5
1428a	Mg	~4000	125.21	90-108	7.28	<5



Thank you Tom King and Robert Sheridan