Jordi Labs

Material Solutions. Uncompromising Integrity.

Elemental Analysis of a Multivitamin Supplement

Released by:

Mark Jordi, Ph.D.
President

Job Number: J12083

Contains No Client Information

200 Gilbert Street
Mansfield, MA 02048
P: (508) 966-1301
jordilabs.com

June 22, 2017

Dear Customer,

Please find enclosed the test results for your sample described as:

1. Men's Multivitamin Tablets

The following tests were performed:

1. Optical Microscopy
2. Scanning Electron Microscopy with Energy-dispersive X-ray Spectroscopy (SEM-EDS)
3. Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Objective

The goal of this analysis was to determine the elemental composition of the vitamin sample using ICP-MS and SEM-EDS. This was performed by analyzing an over-the-counter multivitamin tablet and comparing the results with the manufacturers label claim as well as to limits imposed by *USP <232>*.

Summary of Results

Elemental analysis is a powerful tool for characterizing the metal content of a sample but it is important to use the right tool for the right job. This case study highlights the capabilities of both SEM-EDS and ICP-MS. SEM-EDS correctly identifies and describes the heterogeneous distribution of the multivitamin and provides important information about the varied chemical composition in the tablet. By contrast, the ICP-MS provides extraordinarily accurate quantitative data for the elemental composition of the entire multivitamin tablet but does not provide information about the distribution of the elements.

SEM-EDS Results

One of the multivitamin tablets was divided using a clean scalpel. The split tablet was then mounted onto an SEM stub using carbon tape and imaged optically (**Figure 1**) and with a scanning electron microscope (**Figure 2**) following gold sputter coating to reduce surface charging effects. Optical microscopy provides excellent images however electron microscopy can resolve significantly smaller features down to the micron range. Additionally, the use of Backscatter Electron (BSE) imaging reveals the sample to have several discrete elemental phases, as the back scattered electrons produce a brighter signal for heavier elements.

Elemental mapping was performed on the sample to further distinguish the elemental phases present in the sample. Discrete elemental phases, based on particular EDS histograms, can be mapped across the entire sample, as seen in **Figure 3.** As an example of the sample heterogeneity, consider the elements chlorine and calcium. The label claim for the multivitamin indicates chlorine is present in multiple forms, including potassium chloride and various hydrochloride species (ie: Vitamin B-6). EDS mapping analysis can identify the presence of discrete chlorine-containing compounds and, as seen in **Figure 4**, can also show the co-location of potassium. One can then infer whether a given chlorine-containing spot can be identified as potassium chloride or hydrochloride to corroborate this label claim.

Calcium is another such example, where the element can be found in multiple forms in the vitamin, including calcium carbonate, dicalcium phosphate, calcium pantothenate, and calcium citrate. As seen in **Figure 5**, the dicalcium phosphate groups are easily distinguishable. Notably, the large calcium spots do not appear to correlate to dicalcium phosphate, and likely belong to other calcium sources.

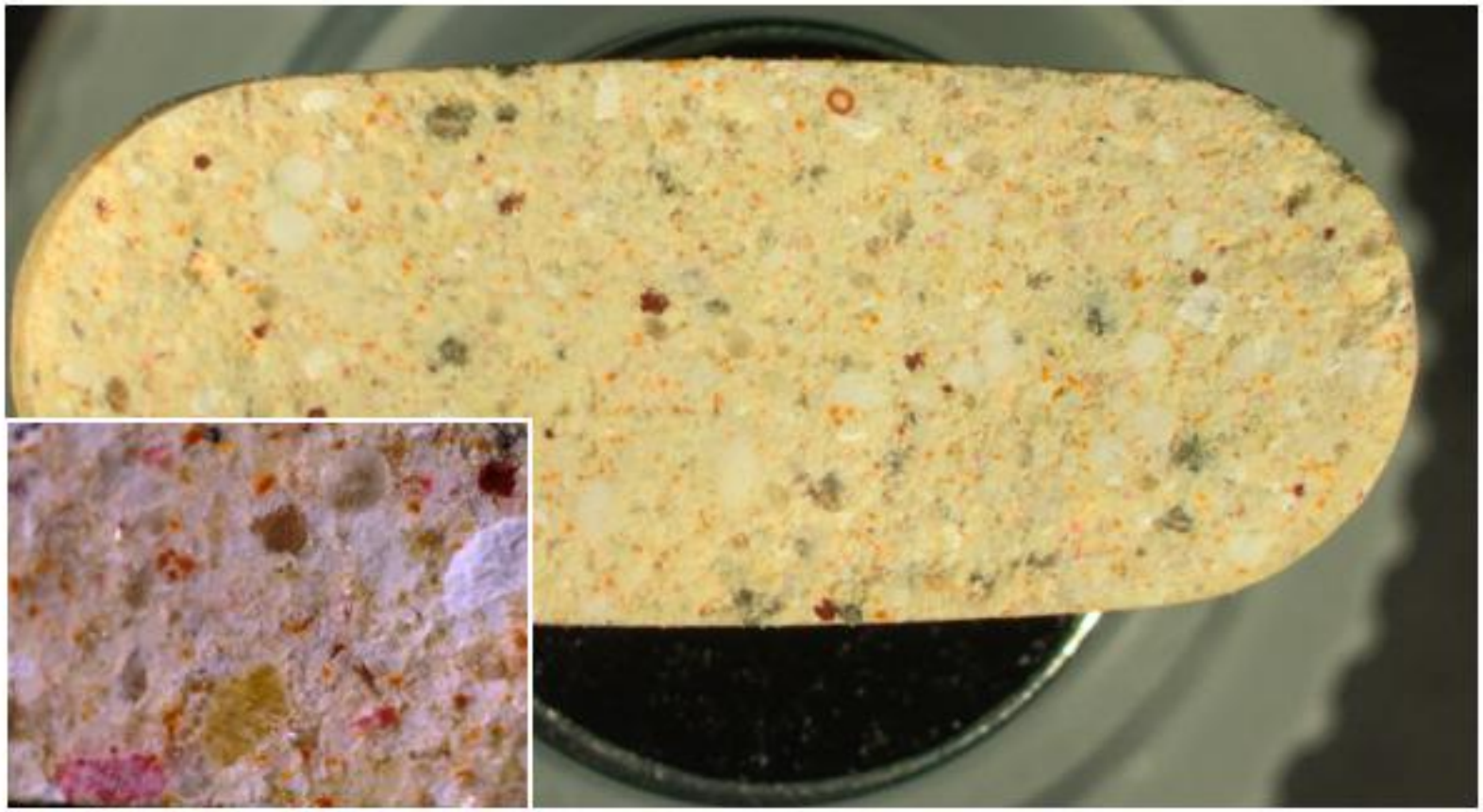

Figure **1**: **Optical microscopy of divided multivitamin along with close up (inset) illustrating inhomogeneous structure of the multivitamin**

Figure 2: Stitched Secondary Electron (SE) images of full multivitamin (Top). Stitched Backscatter Electron (BSE) images of full multivitamin (Bottom)

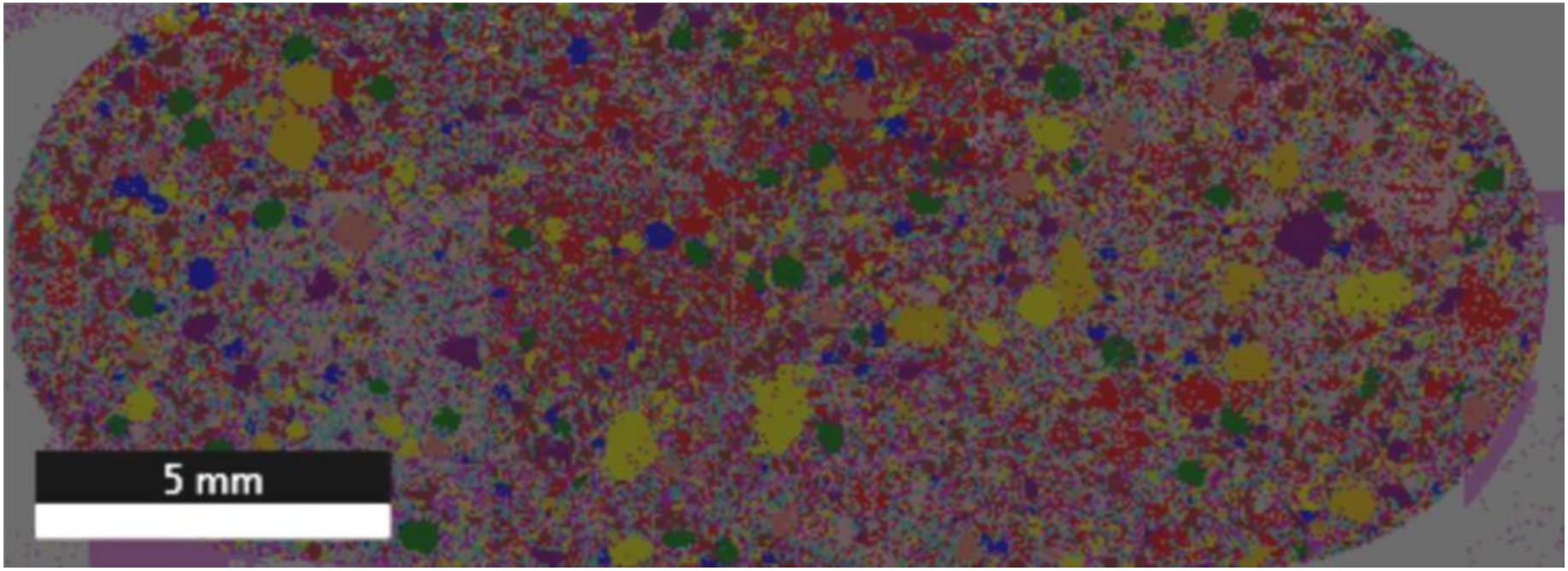

Figure 3: Discrete elemental phases across the divided multivitamin sample. Each color represents a particular series of EDS histogram peaks and can allow distinction of particular compound or mineral.

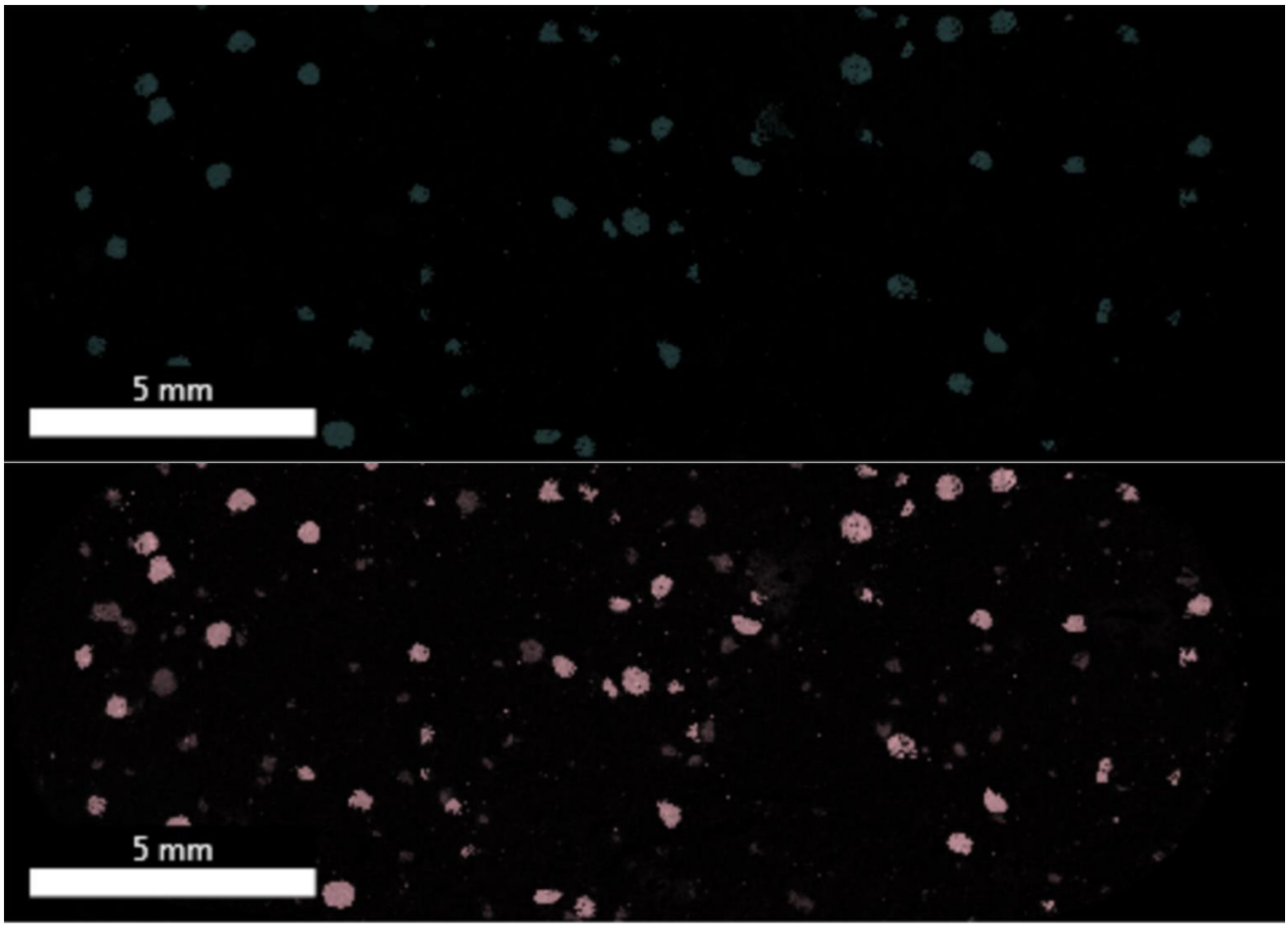

Figure 4: Elemental mapping for Potassium (top, green) and Chlorine (bottom, white).

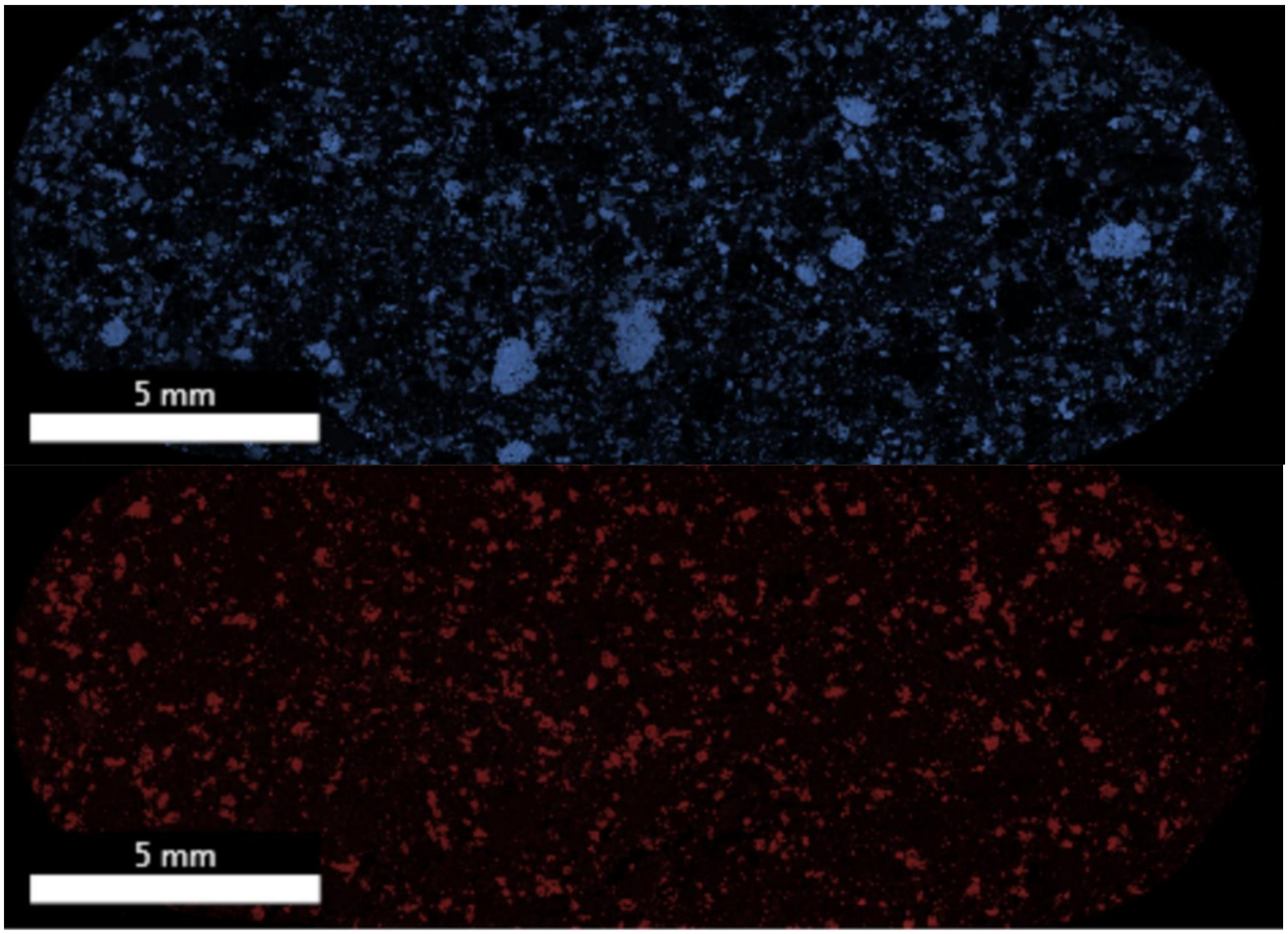

Figure 5: Elemental mapping for Calcium (top, blue) and Phosphorus (bottom, red).

ICP-MS Results

While SEM-EDX can distinguish the distribution of a particular element, a more quantitative approach is required in order to assess the quantitative claims for a multivitamin sample. For such a task, ICP-MS is far better suited.

The sample was weighed and placed into a pre-cleaned Teflon or quartz microwave vessel with 5 mL of concentrated trace metal grade nitric acid. The sample was heated by microwave to 240 °C for 30 minutes. After digestion the sample was brought to a final volume of 50 mL with 2% HNO_3 aqueous solution for Zn, Li, Cr, Be, Al, Co, Ni, and Cd, Se; and 2% HNO_3 and 0.5% HCl aqueous solution for all other elements. An acid blank was also prepared and analyzed to correct for background and possible interferences. All elements analyzed for were calibrated with a minimum calibration coefficient of 0.999. Sample spikes were analyzed with a minimum spike recovery of ± 10%.

The measured amount of each element determined along with a comparison of the manufacturer's indicated amount on the label, and the permissible daily exposures are listed in **Table 1** and **Table 2**. The detailed spike and sample results are listed in **Table 3** and **Table 4**.

Table 1. ICP-MS results on labeled elements (mg/day)

Element	Measured Daily dose	Labeled daily dose	Oral Daily Dose PDE*
Mg	121	100	N.A.
Ca	334	295	
Zn	25	25	
K	106	100	
Mn	2	2	
Cu	2	2	3
V	0.01	0.01	0.1
Cr	0.2	0.2	11
Se	0.277	0.20	N.A.
Mo	0.08	0.08	3

*PDE (permissible daily exposures) data are obtained from *USP <232>*
N.A. - Not Applicable

Table 2. ICP-MS results on elements unlabeled (µg/day)		
Element	**Measured Daily dose**	**Oral Daily Dose PDE ***
Co	5.67	N.A.
Al	416	
Ni	2.26	200
As	0.27	15
Cd	0.33	5
Ba	5.16	N.A.
Be	0.01	
Li	0.48	
Na	197	
Fe	273	
*PDE (permissible daily exposures) data are obtained from *USP <232>* *N.A. - Not Applicable*		

Table 3. ICP-MS results											
Element	**Acid Blank concentration (ng/mL)**	**Sample concentration (ng/mL)**			**Sample concentration in the tablets ($mg/g_{tablets}$)**				**Measured Daily dose (mg/day)**	**Labeled daily dose (mg/day)**	**Percent Difference**
		1	**2**	**3**	**1**	**2**	**3**	**Average**			
Mg	42	364710	291362	336867	36.63	29.53	33.96	33.37	120.94	100	20.9
Ca	347	957521	836214	947557	96.19	84.74	95.52	92.15	333.93	295	13.2
Zn	12	66525	80012	62017	6.68	8.01	6.25	6.98	25.30	25	1.2
K	542	315167	283318	273817	31.6	28.71	27.60	29.32	106.26	100	6.3
Mn	0.02	5495	5448	6130	0.552	0.5455	0.618	0.572	2.07	2	3.6
Cu	0.174	5081	5172	5541	0.510	0.524	0.558	0.531	1.92	2	3.8
V	0.04	37.18	38.50	38.80	0.00374	0.00390	0.00391	0.00385	0.01395	0.01	39.5
Cr	0.01	556.10	777.83	588.54	0.0559	0.07788	0.05933	0.06436	0.233	0.20	16.6
Se	0.009	727.58	817.33	741.09	0.0731	0.08183	0.07471	0.07654	0.277	0.20	38.7
Mo	0.311	216.98	231.66	238.53	0.0218	0.02348	0.02405	0.02311	0.0837	0.075	11.6
Co	0.00	14.09	18.14	14.51	0.00142	0.00182	0.00146	0.00156	0.00567	No data	
Al	1.78	1017	1416	994	0.102	0.1418	0.1002	0.1148	0.416	No data	
Ni	0.00	5.95	6.94	6.21	0.00060	0.00070	0.00063	0.00064	0.00232	No data	
As	0.07	0.70	0.79	0.69	0.00007	0.00008	0.00007	0.00007	0.00027	No data	
Cd	0.00	0.80	1.07	0.83	0.00008	0.00011	0.00008	0.00009	0.00033	No data	
Ba	0.083	16.00	13.50	12.87	0.00161	0.00137	0.00130	0.00142	0.00516	No data	
Be	0.012	0	0	0	0	0	0	0	0	No data	
Li**	0.067	0.56	2.33	1.03	0.00006	0.00023	0.00010	0.00013	0.00048	No data	
Na	9.642	530.92	549.85	536.18	0.0533	0.0557	0.0540	0.0543	0.197	No data	
Fe	0.892	712.68	765.93	763.87	0.0716	0.0776	0.0770	0.0754	0.273	No data	

* Sample concentration in the tablets ($mg/g_{tablets}$)= [sample concentration (ng/mL) * dilution factor (50)/10^6(ng/mg)]/tablet weight

- Tablet weight 1 = 0.4977 g, tablet weight 2 = 0.4934 g for all elements except Zn, Li, Cr, Be, Al, Mn, Co, Ni. Cd and Se for which tablet weight *2* = 0.4994 g, and tablet weight 3 = 0.4960 g.

Measured Daily dose (mg/day) = Average sample concentration in the tablets ($mg/g_{tablets}$)*Tablet mass per day (3.62367 g/day)

Percent Difference = (Measured Daily dose (mg/day) - Labeled Daily dose (mg/day))/ Labeled Daily dose (mg/day)

** This element shows increased variability due to environmental sources at trace levels.

Table 4. ICP-MS spike results						
Element	**Mass**	**spike concentration (ng/mL)**	**Sample concentration (ng/mL)**	**Sample contribution (ng/mL)**	**Spike recovery (ng/mL)**	**Recovery %**
Mg	24	540440	364710	273530	266910	106.76
Ca	43	993100	957520	718140	274960	109.99
Mn	55	1062.57	5448	160.05	902.5	90.25
Zn	67	3370	80012	2350	1020.16	102.02
K	41	350660	273820	222480	128190	102.55
Fe	56	808.09	712.68	705.55	102.53	102.53
Cr	53	1811	777.83	770.05	1033.1	103.3
Mo	95	311.89	219.75	217.55	94.34	94.34
Cu	63	5134.04	5080.67	5029.86	104.18	104.18
Mo	97	312.31	216.98	214.81	97.5	97.5
V	51	130.24	38.5	38.11	92.12	92.12
Co	59	1101.9	18.14	17.96	1083.8	108.4
Ni	60	993.73	6.95	6.88	986.78	98.7
As	75	94.57	0.79	0.78	93.78	93.78
Se	77	1907.29	817.33	809.16	1089.95	108.99
Cd	111	1006.8	1.07	1.06	1005.82	100.6
Al	27	2514	1416.63	1402.4	1097.4	109.7
Be	9	955.2	0	0	955.2	95.5
Li	7	102.36	2.33	2.31	100.02	100.02
Na	23	628.45	536.18	530.82	97.63	97.63

* For Li, Cr, Be, Al, Co, Ni. Cd and Se, Sample contribution = sample concentration
For the remaining elements, Sample Contribution = [sample concentration in ppb * sample volume]/Final volume
Sample volume = 6 mL for Mg and Ca; 6.5 mL for K, 1 mL for Mn and Zn; 4950 µL for other elements;
Final volume = 8 mL for Mg, Ca, K, 34.04 mL for Mn and Zn, 5 mL for other elements
- Recovery = (Measured Conc. - Sample Contribution)/Spike amount
- Spike amount = 250 µg/mL for Mg and Ca, 125 ppm for K, 1µg/mL for Zn, Cr, Mn, Co, Ni, Be, Se, Cd, Al, 100 ng/mL for Fe, Mo, Cu, V, As, Na and Li

Analysis Conditions

This section of a Jordi report provides information on the methods used including instrument type, temperatures, solvents, sample preparation, etc. The specific conditions have been removed for this case study.

Closing Comments

Jordi Labs' reports are issued solely for the use of the clients to whom they are addressed. No quotations from reports or use of the Jordi name is permitted except as authorized in writing. The liability of Jordi Labs with respect to the services rendered shall be limited to the amount of consideration paid for such services and do not include any consequential damages.

Jordi Labs specializes in polymer testing and has 30 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

Leland Martin

Leland Martin, M.S.
Senior Chemist
Jordi Labs LLC

James Woods

James Woods, Ph. D.
Senior Research Scientist
Jordi Labs LLC

Lan Luo

Lan Luo, Ph.D.
Senior Chemist
Jordi Labs LLC

Mark Jordi

Mark Jordi, Ph. D.
President
Jordi Labs LLC