ColdBlock™ Technologies Inc.

APPLICATION NOTE

AMIS 0656 – Spodumene Concentrate, Brazil

SUMMARY

The application note summarizes the digestion of AMIS 0656, a Spodumene Concentrate Certified Reference Material using ColdBlock™ Digestion Pro Series Technology.

Instrument:	ColdBlock CBM sample digester, chiller, HF compatible liners, ICP-MS & ICP-OES
Published:	June 2023
Digestion Time:	30 Minutes
Acid Used:	Aqua Regia, HF & $H3BO_3$
Average ColdBlock Recovery vs. CRM:	■ 100% Lithium ■ 105% Tantalum ■ 91% Niobium

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 0.25g of each sample was weighed and placed into a ColdBlock™ Digestion vessel
3. 20 mL of Aqua Regia + 5mL HF was added
4. Sample was digested at 80% power for 20 minutes
5. 20mL of 10% $_{w/v}$ Boric acid was added
6. Samples were digested again at 80% power for 10 minutes
7. Samples were cooled and bulked to 50mL using 2% HNO_3 + 0.5% HCl $_{v/v}$

DISCUSSION

- The addition of Boric acid will help re-solubilize any insoluble fluorides and will help neutralize any remaining HF in solution
- Samples appeared mostly clear at the end of the digestion; a minor amount of material settled on the bottom of the tube after bulk up

This material was made using ore sourced from the Volta Grande mine in Minas Gerais state, Brazil.

AMIS0656; Spodumene Concentrate, Brazil; AMIS matrix Reference Materials; A Division of Torre Analytical Services; Gauteng, South Africa (May, 2019)

AMIS 0656– Spodumene Concentrate, Brazil

Results

AMIS 0656– Spodumene Concentrate, Brazil											
Method:	**0.25g**	**20mL Aqua Regia + 4mL HF, 80% 20 minutes – add 20mL 10% $_{w/v}$ Boric acid, 80% 10 minutes**									
Element	**Certified Value (ppm)**	**+/-**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
			Low	**High**							
***Ag**	0.1	N/A	N/A	N/A	0.10	0.09	0.08	0.1	0.01	12.67%	91%
***Al**	120702.9	N/A	N/A	N/A	131690	129711	126140	129180.3	2812.9	2.18%	107%
As	2	1	1	3	2.0	2.0	2.0	2	0.03	1.60%	100%
Ba	17	2	15	19	18	18	18	18	0.3	1.74%	105%
Be	273	45	228	318	268.7	248.9	254.9	258	10.2	3.94%	94%
Bi	2	0.5	1.5	2.5	3.40	3.20	3.50	3	0.2	4.54%	168%
***Ca**	7713.6	N/A	N/A	N/A	7577	7866	7005	7482.5	438.1	5.86%	97%
***Cd**	0.3	N/A	N/A	N/A	0.3	0.3	0.4	0.3	0.01	1.68%	114%
***Ce**	4.3	N/A	N/A	N/A	5.4	5.4	5.3	5.4	0.1	1.08%	125%
Co	3	0.5	2.5	3.5	3.17	3.18	3.03	3	0.1	2.68%	104%
***Cr**	99.9	N/A	N/A	N/A	128.0	127.4	124.0	126.5	2.1	1.69%	127%
Cs	86	23	63	109	94.06	96.63	95.44	95	1.3	1.35%	111%
Cu	25	6	19	31	28.2	25.6	23.9	26	2.2	8.40%	104%
***Fe**	8769.6	N/A	N/A	N/A	9349	9129	9034	9170.5	161.6	1.76%	105%
Ga	140	9	131	149	147	148	146	147	1.2	0.81%	105%
Hf	7	2	5	9	7	7	7	7.1	0.4	5.95%	102%
K	3262	326	2936	3588	3274	3208	3262	3248	34.9	1.08%	100%
***La**	1.7	N/A	N/A	N/A	1.9	2.0	1.9	1.9	0.1	3.13%	113%
Li	29400	2900	26500	32300	29894	29635	28784	29438	580.5	1.97%	100%
***Mg**	384.7	N/A	N/A	N/A	583.4	569.7	572.2	575.1	7.3	1.27%	149%
Mn	1006	102	904	1108	1078	1046	1039	1055	20.8	1.97%	105%
Mo	1	0.1	0.9	1.1	1.35	1.23	1.27	1	0.1	4.76%	128%
Na	3879	246	3633	4125	4051	3918	3879	3949	89.8	2.27%	102%
Nb	26	5	21	31	24.38	22.94	23.88	24	0.7	3.08%	91%
Ni	16	5	11	21	17	17	19	18	0.9	5.21%	109%
P	1427	189	1238	1616	1624	1517	1507	1550	64.9	4.19%	109%
Pb	24	4	20	28	26.7	27.3	27.7	27	0.5	1.75%	113%
***Rb**	838.7	N/A	N/A	N/A	993.2	988.2	978.4	986.6	7.5	0.76%	118%
****Si**	297200	N/A	N/A	N/A	273608	265714	267564	268962	4128.6	1.54%	90%
Sb	4	0.6	3.4	4.6	4.14	4.00	4.04	4	0.1	1.78%	102%
***Sn**	156.5	N/A	N/A	N/A	153.3	143.2	150.3	148.9	5.2	3.50%	95%

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Results

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Element	**Certified Value (ppm)**	**+/-**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
			Low	**High**							
***Sr**	22.5	N/A	N/A	N/A	22.8	23.3	22.4	22.8	0.5	2.06%	102%
Ta	187	35	152	222	191.81	199.61	198.87	197	4.3	2.19%	105%
***Th**	3.7	N/A	N/A	N/A	4.87	4.36	4.3	4.5	0.3	6.94%	122%
Ti	305	20	285	325	295	294	282	290	7.1	2.46%	95%
Tl	7	0.7	6.3	7.7	7.92	8.02	8.06	8	0.1	0.90%	114%
***U**	2.8	N/A	N/A	N/A	5.02	5.07	5.19	5.1	0.1	1.72%	182%
V	8	0.9	7.1	8.9	7.2	7.0	6.8	7	0.2	2.44%	87%
W	1	0.4	0.6	1.4	1.4	1.4	1.4	1	0.0	1.89%	140%
***Y**	6.7	N/A	N/A	N/A	10.36	10.08	9.67	10	0.3	3.46%	150%
Zn	58	6	52	64	60	58	58	59	1.0	1.69%	101%
Zr	32	8	24	40	34.84	33.19	34.08	34	0.8	2.43%	106%

* Represents uncertified elements
**Represents elements certified by Peroxide Fusion (no 4-acid data available)

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