

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 & H ₃ BO ₃
Average ColdBlock Recovery vs. CRM:	<ul style="list-style-type: none">■ 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 + 4mL HF was added
4. Sample was digested at 80% power for 20 minutes
5. 20mL of 4%_{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% HNO₃ + 0.5% HCl_{v/v}

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)

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

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Results

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Method:	0.25g	20mL Aqua Regia + 4mL HF, 80% 20 minutes - add 20mL 4% _{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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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)