ColdBlock™ Technologies Inc.

APPLICATION NOTE

Lead Concentrate, AMIS 0618

SUMMARY

The application note summarizes the digestion of AMIS 0618, a lead concentrate certified reference material using ColdBlock™ Digestion Pro Series Technology.

Instrument:	ColdBlock CBM Pro-Series sample digester, chiller, HF liners, ICP-OES
Published:	March 2025
Digestion Time:	30 Minutes
Acid Used:	HNO_3, HCl, HF, & H_3BO_3
Average ColdBlock Recovery vs. CRM:	■ 100% antimony ■ 96% arsenic ■ 101% lead

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. 20mL of reverse aqua regia + 3mL HF was added
4. Samples were digested at 80% power for 20 minutes
5. 20mL of 4% boric acid $_{v/v}$ 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

DISCUSSION

- The addition of Boric acid will help re-solubilize any insoluble fluorides and will help neutralize any remaining HF in solution
- After digestion, the samples were clear, and no visible sample material remained
- To improve silver recoveries, bulk samples in >20% HCl (precipitation of insoluble lead chloride ($PbCl_2$) might occur)

AMIS 0618 after bulk up in 2% HNO_3

The material for AMIS 0618 was provided by Black Mountain Mining and is a lead concentrate made from Black Mountain Broken Hill type ore from the Northern Cape Province in South Africa www.Amis.co.za

AMIS 0618; Lead Concentrate, Black Mountain, Northern Cape, South Africa; AMIS, Matrix Reference Materials; Gauteng, South Africa (May 2019)

Results

AMIS 0618, Lead Concentrate						
Method:	0.25g – Add 20mL reverse aqua regia (1:3, HCl:HNO_3) + 3mL HF and digest at 80% power for 20 minutes. Add 20mL of 4% boric acid $_{v/v}$ and digest again at 80% power for 10 minutes. Let cool, and bulk to a final volume of 50mL with 2% HNO_3					
Element	AMIS Certified 4-Acid Value (mg/kg)	AMIS +/-	ColdBlock Average (mg/kg) n=5	ColdBlock Stdev	ColdBlock % RSD	ColdBlock % Recovery
Antimony	175	38	175	7.42	4.2%	100%
Arsenic	212	24	204	8.72	4.3%	96%
Barium	97	14	102	0.79	0.8%	105%
Bismuth	2863	229	2819	57.56	2.0%	99%
Cadmium	88	8	90	1.60	1.8%	102%
Calcium	939	212	846	9.31	1.1%	90%
Cobalt	228	17	246	2.52	1.0%	108%
Copper	20100	1200	20146	259.09	1.3%	100%
Iron	51900	3200	51344	761.66	1.5%	99%
Lead	698600	8200	702116	10184	1.5%	101%
Magnesium	1193	173	1135	15.82	1.4%	95%
Manganese	6551	388	6209	74.45	1.2%	95%
Molybdenum	221	34	248	4.85	2.0%	112%
Nickel	73	8	83	2.93	3.6%	113%
Silver	592	53	260	54.4	21%	44%
Sulfur*	141600	3500	148154	2633.70	1.8%	105%
Strontium	3	0.8	3	0.03	0.8%	101%
Zinc	19800	610	19895	333.46	1.7%	101%

*Sulfur certified by combustion/leco

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