ColdBlock™ Technologies Inc.

APPLICATION NOTE

Lead Concentrate, CPb-3

SUMMARY

The application note summarizes the digestion of CPb-3, a lead concentrate certified reference material using ColdBlock Digestion Pro Series Technology.

Instrument:	ColdBlock CBM Pro-Series, chiller, HF liners, ICP-OES
Published:	Mary 2025
Digestion Time:	30 Minutes
Acid Used:	HNO_3, HCl, HF & H_3BO_3
Average ColdBlock Recovery vs. CRM:	■ 100% antimony ■ 102% lead ■ 100% zinc

METHODOLOGY

1. Set the chiller temperature to -5°C.
2. Weigh 0.25 g of each sample and transfer into a ColdBlock Digestion vessel with liner
3. Add 20 mL of reverse aqua regia (add HNO_3 first, followed by HCl) and 3 mL of HF to each vessel.
4. Digest samples at 80% power for 20 minutes.
5. Add 20 mL of 4% boric acid ($_{w/v}$) to each sample.
6. Digest samples again at 80% power for 10 minutes.
7. Cool the samples and adjust the volume to 50 mL with DI water

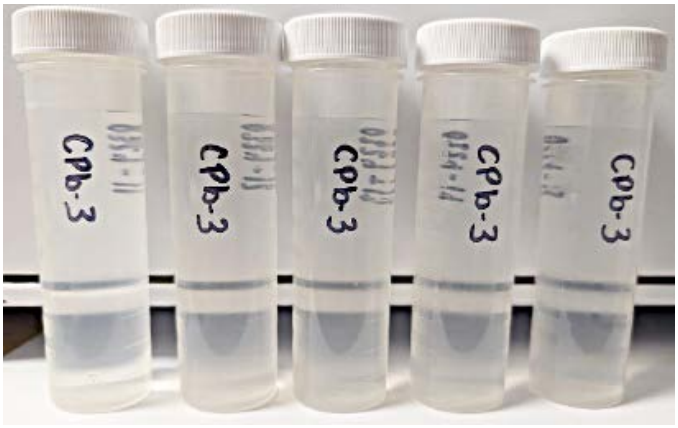

Figure 1- CPb-3 after bulking-up to 50mL

DISCUSSION

- The addition of boric acid aids in the re-solubilization of insoluble fluorides and neutralizes residual hydrofluoric acid (HF). This step is crucial for sample dissolution and minimizing analyte loss during digestion.
- Following the digestion process, the samples were visibly clear - indicating that the sample matrix had dissolved. A trace amount of material settled on the bottom of the tube – see figure 1

Results

CPb-3, Lead Concentrate					
Method:	**0.25g - Add 20mL reverse aqua regia + 3mL HF and digest at 80% power for 20 minutes. Add 20mL of 4% boric acid $_{w/v}$ and digest again at 80% power for 10 minutes. Let cool, and adjust the volume to 50mL with DI water**				
Element	**CPb-3 Certified Values**	**ColdBlock Average n=5**	**ColdBlock Stdev +/-**	**ColdBlock RSD**	**ColdBlock Recovery vs Certified Value**
Al (%)	0.203	0.200	0.005	2.5%	99%
Ca (%)	0.059	0.053	0.003	5.7%	90%
Cd (%)	0.0652	0.0653	0.002	3.1%	100%
Co (µg/g)	13.6	13.7	1.4	10.2%	101%
Cr (%)	0.0102	0.0104	0.0002	1.9%	102%
Cu (%)	0.240	0.245	0.004	1.6%	102%
Fe (%)	8.45	8.51	0.08	0.9%	101%
Mg (%)	0.1062	0.1	0.0024	2.3%	100%
Pb (%)	58.53	59.02	0.39	0.7%	101%
S (%)	17.03	17.93	0.21	1.2%	105%
Sb (%)	0.5800	0.5778	0.008	1.4%	100%
Zn (%)	5.96	5.98	0.04	0.7%	100%

The material for CPb-3 was donated by a North American refinery.

CCRMP CanmetMINING NRCan (2020). Certificate of Analysis for CPb-3, Lead Concentrate https://natural-resources.canada.ca/

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