

Strong Acid Digestion

SUMMARY

The application note summarizes the digestion of a range of certified reference materials by AMIS, Geostats, CDN Lab and OREAS using ColdBlock™ Digestion Pro Series Technology.

Instrument:	ColdBlock Pro Series CBM sample digester, HF compatible liners, chiller, ICP-OES & ICP-MS
Published:	January 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% Arsenic■ 100% Copper■ 102% Silver

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 Aqua Regia and 3mL HF was added
4. Samples were digested at 80% power for 20 minutes
5. 20mL of 10% _{v/v} Boric acid was added
6. Samples were digested again at 80% power for 10 minutes
7. Samples were cooled and bulked to 50mL with 2% HNO₃ + 0.5% HCl _{v/v}

DISCUSSION

- All samples were digested in triplicate
- The addition of Boric acid will help re-solubilize any insoluble fluorides and will help neutralize any remaining HF in solution
- If Silver precipitates out of solution as AgCl, bulk up with >10% HCl _{v/v}
- If the Sulfide content of your sample is > 10 wt.% - reverse the ratios of Aqua Regia and use 1:3, HCl:HNO₃ – always add the Nitric acid first (reddish brown NO₂ fumes might form)

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Results

% Recovery of CRM certified analytes by ColdBlock strong acid digestion method compared to 4-Acid CRM certificate value											
Elements	AMIS CRMs % Recoveries			CDN-LABS CRMs % Recoveries			Geostats CRMs % Recoveries		OREAS CRMs % Recoveries		
	AMIS 0566	AMIS 0559	AMIS 0571	CDN- ME-1709	CDN- ME-1805	CDN- ME-1902	GBMS 304-4	GBM 909-15	OREAS 503C	OREAS 134b	OREAS 901
Ag	100%	116%	N/A	107%	104%	93%	96%	94%	103%	95%	107%
Al	97%	99%	101%	N/A	N/A	N/A	N/A	N/A	97%	95%	99%
As	99%	101%	97%	N/A	N/A	N/A	103%	N/A	99%	105%	95%
Ba	102%	108%	106%	N/A	N/A	N/A	N/A	N/A	115%	N/A	102%
Be	N/A	100%	107%	N/A	N/A	N/A	N/A	N/A	116%	N/A	94%
Bi	98%	117%	123%	N/A	N/A	N/A	N/A	N/A	104%	N/A	97%
Ca	97%	99%	100%	N/A	N/A	N/A	N/A	N/A	102%	94%	108%
Cd	112%	113%	100%	N/A	N/A	N/A	N/A	N/A	97%	100%	N/A
Ce	105%	102%	104%	N/A	N/A	N/A	N/A	N/A	98%	N/A	95%
Co	96%	97%	98%	N/A	N/A	N/A	103%	N/A	97%	96%	100%
Cr	N/A	103%	N/A	N/A	N/A	N/A	N/A	N/A	106%	N/A	110%
Cs	N/A	105%	101%	N/A	N/A	N/A	N/A	N/A	107%	N/A	107%
Cu	99%	101%	101%	100%	98%	102%	102%	98%	97%	99%	100%
Fe	97%	99%	99%	N/A	N/A	N/A	N/A	N/A	98%	98%	98%
Ga	96%	102%	101%	N/A	N/A	N/A	N/A	N/A	96%	N/A	96%
Hf	N/A	88%	113%	N/A	N/A	N/A	N/A	N/A	96%	N/A	96%
K	97%	96%	101%	N/A	N/A	N/A	N/A	N/A	98%	N/A	101%
La	112%	106%	109%	N/A	N/A	N/A	N/A	N/A	100%	N/A	97%
Li	N/A	96%	97%	N/A	N/A	N/A	N/A	N/A	101%	N/A	97%
Lu	N/A	100%	109%	N/A	N/A	N/A	N/A	N/A	96%	N/A	99%
Mg	100%	100%	103%	N/A	N/A	N/A	N/A	N/A	102%	100%	101%
Mn	100%	107%	102%	N/A	N/A	N/A	N/A	N/A	102%	N/A	101%
Na	105%	110%	100%	N/A	N/A	N/A	N/A	N/A	86%	N/A	90%

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Ni	N/A	95%	104%	N/A	N/A	N/A	106%	102%	106%	N/A	98%
Pb	98%	106%	109%	112%	100%	104%	118%	102%	98%	102%	98%
Rb	107%	96%	95%	N/A	N/A	N/A	N/A	N/A	96%	N/A	101%
Sb	106%	106%	106%	N/A	N/A	N/A	N/A	N/A	109%	98%	98%
Sc	N/A	105%	88%	N/A	N/A	N/A	N/A	N/A	106%	N/A	109%
Se	N/A	97%	N/A	N/A	N/A	N/A	N/A	N/A	92%	N/A	N/A
Sn	N/A	88%	88%	N/A	N/A	N/A	N/A	N/A	79%	N/A	87%
Sr	99%	94%	94%	N/A	N/A	N/A	N/A	N/A	96%	N/A	96%
Te	100%	97%	N/A	N/A	N/A	N/A	N/A	N/A	127%	N/A	124%
Th	99%	114%	117%	N/A	N/A	N/A	N/A	N/A	98%	N/A	100%
Tl	N/A	96%	122%	N/A	N/A	N/A	N/A	N/A	107%	N/A	107%
Tm	115%	117%	100%	N/A	N/A	N/A	N/A	N/A	99%	N/A	N/A
U	104%	103%	104%	N/A	N/A	N/A	N/A	N/A	110%	N/A	100%
V	98%	108%	91%	N/A	N/A	N/A	N/A	N/A	101%	N/A	99%
Y	98%	87%	94%	N/A	N/A	N/A	N/A	N/A	92%	N/A	96%
Yb	N/A	133%	118%	N/A	N/A	N/A	N/A	N/A	98%	N/A	98%
Zn	99%	98%	101%	103%	101%	103%	99%	100%	104%	98%	104%
Zr	N/A	92%	88%	N/A	N/A	N/A	N/A	N/A	85%	N/A	99%