

APPLICATION NOTE

Contaminated Soil, SS-1

SUMMARY

The application note summarizes the digestion of SS-1 a contaminated soil Certified Reference Material using ColdBlock™ Digestion Pro Series Technology.

Instrument: ColdBlock CBM sample digester, chiller, ICP-OES, ICP-MS (with CRC technology)

Published: December 2024

Digestion Time: 45 minutes

Acid Used: HNO₃, HCl & H₂O₂

Average ColdBlock Recovery vs. CRM:

- 99% Antimony
- 96% Silver
- 101% Lead

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 0.5g of each sample was weighed and placed into a ColdBlock™ Digestion vessel
3. 10mL of 1:1 HNO₃ + 5mL 1:1 HCl was added, and the samples were digested at 70% power for 5 minutes
4. 10mL HNO₃ was added and digested again at 60% power for 20 minutes (repeat this step if fuming continues)
5. 3mL of 30% H₂O₂ was added slowly, and digested again at 60% for 10 minutes
6. Add 5mL HCl and digest a final time at 60% power for 10 minutes
7. Samples were cooled and bulked to 50mL using ultrapure water



Contaminated Soil, SS-1 after bulk up to 50mL

DISCUSSION

- This method yielded data comparable to EPA 3050b standards (with improved recovery of antimony) in just 45 minutes.
- The addition of HCl in the final step of the digestion helps keep certain elements such as antimony and silver solubilized
- This method is not appropriate for standard ICP-MS instruments that are not equipped with Collision/Reaction Cell technology, for more detailed information please contact ColdBlock at info@coldblock.ca

Results

Contaminated Soil, SS-1											
Method:	0.5g	10mL 1:1 HNO ₃ + 5mL 1:1 HCl, digest at 70% for 5 minutes, then add 10mL HNO ₃ and digest at 60% for 20 minutes (repeat this step if fuming continues) Add 3mL of 30% H ₂ O ₂ , slowly and digest again @ 60% for 10 minutes, then add 5mL HCl and digest again @ 60% for 10 minutes. Cool and bulk to 50mL with 2% HNO ₃									
Element	Certified Value	+/-	Sample A	Sample B	Sample C	Sample D	Sample E	Average (ppm)	Stdev	% RSD	% Recovery vs certified value
Ag	0.88	0.03	0.84	0.84	0.89	0.84	0.82	0.85	0.03	3.1%	96%
Al	12163	410	12077	12634	11519	11542	11985	11951	458	3.8%	98%
As	20.7	1	19.3	20.6	19.6	19.9	20.3	19.9	0.522	2.6%	96%
Ba	464	16	445	473	456	431	436	448	16.8	3.8%	97%
Be	0.48	0.05	0.49	0.46	0.47	0.48	0.49	0.48	0.013	2.7%	100%
Ca	50265	1213	49491	51721	47580	47290	49195	49055	1775	3.6%	98%
Cd	3.2	0.2	3.5	3.4	3.2	3.4	3.0	3.3	0.20	6.1%	103%
Co	12.9	0.4	12.8	13	12.4	12.1	12.9	12.6	0.38	3.0%	98%
Cr	103	5	98.1	103	101	97.5	98.8	100	2.28	2.3%	97%
Cu	403	10	399	427	384	370	385	393	21.6	5.5%	98%
Fe	72000	2273	69216	71857	67438	68507	68635	69131	1654	2.4%	96%
Hg	0.41	0.02	0.38	0.40	0.39	0.41	0.38	0.39	0.01	3.3%	96%
K	2232	150	2493	2576	2438	2523	2468	2500	53	2.1%	112%
Mg	9690	230	9275	9325	9206	9274	9547	9325	131	1.4%	96%
Mn	737	19	708	738	691	687	719	709	21	3.0%	96%
Mo	6.8	0.3	7.3	7.4	6.5	7.5	6.9	7.1	0.41	5.8%	105%
Na	650	64	637	641	582	593	615	614	26.1	4.3%	94%
Ni	59.2	1.3	59.8	62.1	59.2	56.5	57.1	58.9	2.24	3.8%	100%
Pb	764	15	764	794	784	753	767	772	16.4	2.1%	101%
Sb	5.5	1.1	5.6	5.2	6	5.3	5.2	5.5	0.34	6.3%	99%
Se	0.78	0.14	0.82	0.86	0.78	0.8	0.71	0.79	0.055	7.0%	102%
V	27.2	1.4	26.6	26.9	26.1	26.0	26.0	26.3	0.409	1.6%	97%
Zn	1114	37	1130	1179	1156	1095	1100	1132	36	3.2%	102%