

APPLICATION NOTE

Industrial Sludge, NIST 2782

SUMMARY

The application note summarizes the digestion of NIST 2782, an Industrial Sludge standard reference material using ColdBlock Digestion Pro Series Technology.

Instrument:	ColdBlock CBM, chiller, ICP-OES, ICP-MS (with CRC technology)
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Digestion Time:	45 Minutes
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Acid Used:	HNO ₃ , HCl & H ₂ O ₂
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Average ColdBlock Recovery vs. CRM:	<ul style="list-style-type: none">■ 109% Arsenic■ 103% Cadmium■ 102% Antimony
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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 were 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. 5mL HCl was added and digested at 60% power for 10 minutes
7. Samples were cooled and bulked to 50mL using ultrapure water.



15 samples of NIST 2782 after bulk up to 50mL

DISCUSSION

- This method yielded data comparable to EPA 3050B standards 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

Results

Industrial Sludge, NIST 2782					
Method:	0.5g - Add 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 ultrapure water				
Element	*Reference Value (mg/kg) n=5	ColdBlock Average (mg/kg) n=16	ColdBlock Stdev	ColdBlock % RSD	ColdBlock % Recovery vs SW-846 3050B
Arsenic	144	157	3.19	2.2%	109%
Aluminum	1468	1503	41.12	2.8%	102%
Antimony	2.07	2.12	0.420	20%	102%
Barium	136	153	2.14	1.6%	113%
Cadmium	2.14	2.20	0.112	5.3%	103%
Calcium	4544	4624	169.6	3.7%	102%
Chromium	65.4	69.4	1.46	2.2%	106%
Cobalt	52.2	53.5	1.86	3.6%	103%
Copper	2288	2511	66.06	2.9%	110%
Iron	247576	258116	7041	2.8%	104%
Lead	491	553	5.69	1.2%	113%
Magnesium	468	482	23.8	5.1%	103%
Manganese	243	264	8.64	3.6%	109%
Mercury	1.22	1.27	0.0509	4.2%	104%
Molybdenum	5.39	5.95	0.374	6.9%	110%
Nickel	95.54	106.2	4.65	4.9%	111%
Potassium	88	97	6.1	7.0%	111%
Silver	26.98	28.47	3.212	11.9%	106%
Sodium	2539	2637	75.98	3.0%	104%
Vanadium	10.7	14.4	0.678	6.3%	135%
Zinc	1143	1129	46.91	4.1%	99%