

APPLICATION NOTE

Metals in Sewage Sludge

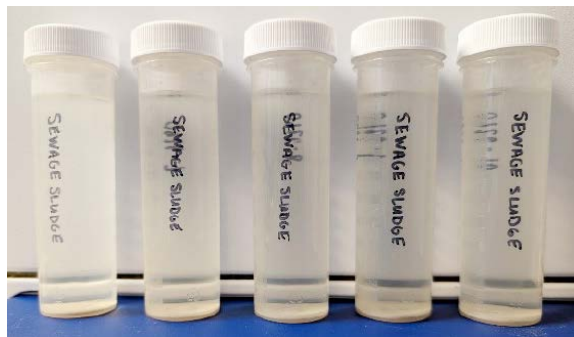
SUMMARY

The application note summarizes the digestion of SQC001S, a sewage sludge 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:	HNO ₃ , HCl & H ₂ O ₂
Average ColdBlock Recovery vs. CRM:	<ul style="list-style-type: none">■ 103% Arsenic■ 96% Mercury■ 98% 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



Metals in Sewage Sludge, CRM, SQC001S, 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

Metals in Sewage Sludge											
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 5.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 (ppm)	42.3	5.5	37.2	38.4	37.4	41.2	44.2	39.7	2.99	7.5%	94%
Al (ppm)	18800	3120	17907	19519	18208	17571	18164	18274	741	4.1%	97%
As (ppm)	282	35.9	284	294	293	271	316	292	16.5	5.6%	103%
Ba (ppm)	430	40.7	425	449	435	389	412	422	22.9	5.4%	98%
Be (ppm)	45.1	4.8	48.9	48.5	48.8	48.8	48.1	48.6	0.327	0.7%	108%
Ca (ppm)	66200	8280	68389	71253	65949	67212	67194	67999	2013	3.0%	103%
Cd (ppm)	64.4	5.2	59	60.5	62.9	62.9	65.6	62.2	2.53	4.1%	97%
Co (ppm)	49.3	4.39	48.4	47.4	45.9	48.6	53	48.7	2.65	5.4%	99%
Cr (ppm)	116	14.6	121	119	121	123	137	124	7.29	5.9%	107%
Cu (ppm)	416	33.8	416	391	396	423	485	422	37.6	8.9%	101%
Fe (ppm)	33900	5630	31232	32641	32119	31724	36715	32886	2202	6.7%	97%
Hg (ppm)	6.06	1.44	5.91	5.62	5.91	5.77	5.96	5.83	0.139	2.4%	96%
K (ppm)	4330	161	3991	3968	4447	4062	4484	4190	254	6.1%	97%
Mg (ppm)	13200	1110	14243	14001	13618	13184	14129	13835	433	3.1%	105%
Mn (ppm)	1310	350	1130	1550	1191	1340	1246	1291	164	12.7%	99%
Mo (ppm)	102	17	95.2	96.6	103.6	107.5	107.6	102	5.91	5.8%	100%
Na (ppm)	3100	304	2895	2967	2783	2995	3066	2941	107.6	3.7%	95%
Ni (ppm)	106	6.42	103.2	108.5	106.4	104	109.7	106	2.80	2.6%	100%
Pb (ppm)	126	10.5	121	121	125	119	130	123	4.38	3.6%	98%
Sb (ppm)	70.8	24	66.3	66.8	69.1	69.0	75.9	69.4	3.84	5.5%	98%
Se (ppm)	146	18.6	137	144	151	154	145	146	6.61	4.5%	100%
Tl (ppm)	40.9	10.9	38.2	39.1	38	37.7	44.1	39.4	2.67	6.8%	96%
V (ppm)	105	15.8	104	104	96.8	92.8	107	101	5.89	5.8%	96%
Zn (ppm)	913	82.8	905	885	863	881	1002	907	55.1	6.1%	99%