ColdBlock™ Technologies Inc.

APPLICATION NOTE

Estuarine Sediment, NIST 1646a

SUMMARY

The application note summarizes the digestion of NIST 1646a, an Estuarine Sediment standard reference material using ColdBlock Digestion Pro Series Technology.

Instrument:	ColdBlock CBM, chiller, ICP-OES, ICP-MS (with CRC technology)
Published:	March 2025
Digestion Time:	45 Minutes
Acid Used:	HNO_3, HCl & H_2O_2
Average ColdBlock Recovery vs. CRM:	■ 102% Barium ■ 107% Chromium ■ 102% Molybdenum

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 HNO_3 + 5mL 1:1 HCl were added, and the samples were digested at 70% power for 5 minutes
4. 10mL HNO_3 was added and digested again at 60% power for 20 minutes (repeat this step if fuming continues)
5. 3mL of 30% H_2O_2 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 1646a 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

Estuarine Sediment, NIST 1646a					
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_2O_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
Aluminum	7075	7182	206	2.9%	102%
Antimony	0.72	0.81	0.04	5.1%	112%
Arsenic	4.69	6.12	0.28	4.5%	130%
Barium	21.2	21.7	1.03	4.7%	102%
Cadmium	0.127	0.142	0.00	2%	112%
Calcium	3578	3711	162	4.4%	104%
Chromium	24.4	26.2	2.15	8.2%	107%
Cobalt	3.2	3.4	0.13	3.8%	108%
Copper	9.98	9.96	0.13	1.3%	100%
Iron	15330	17111	849	5.0%	112%
Lead	5.26	4.99	0.3	5.1%	95%
Magnesium	2972	3347	35	1.1%	113%
Manganese	117.3	125.2	1.7	1.3%	107%
Mercury	0.03	0.05	0.0039	8.5%	142%
Molybdenum	1.81	1.84	0.15	8.4%	102%
Nickel	18.8	21.9	2.27	10.4%	116%
Potassium	2235	2273	96	4.2%	102%
Sodium	3819	3913	254	6.5%	102%
Vanadium	20.95	22.90	1.10	4.8%	109%
Zinc	36.72	35.92	2.06	5.7%	98%

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