

Metals in Sewage SludG™, ERA 160

SUMMARY

The application note summarizes the digestion of ERA 160, a Metals in Sewage Sludge certified 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 ₂ O ₂
Average ColdBlock Recovery vs. CRM:	107% Arsenic103% Mercury105% Selenium

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% $\rm H_2O_2$ was added slowly, and digested again at 60% for 10 minutes
- 6. 5mL HCl was added and digested at 60% power for 10 minutes
- Samples were cooled and bulked to 50mL using ultrapure water



15 samples of ERA 160 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

ERA #160 Metals in Sewage SludG (lot:D122-160) 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 Method: ColdBlock ColdBlock % *Reference Value ColdBlock ColdBlock Element Average (mg/kg) Recovery vs % RSD (mg/kg) n=5Stdev SW-846 3050B n=16 Aluminum 3228 42.10 1.2% 106% 3427 108 124 4.16 3.4% 115% Antimony 1.89 107% Arsenic 127 136 1.4% 718 103% **Barium** 698 23.6 3.3% Beryllium 78.3 80.5 2.90 3.6% 103% Cadmium 86.7 85.4 2.71 3.2% 99% Calcium 39519 40469 1524.6 3.8% 102% Chromium 72.4 74.7 103% 1.72 2.3% 9.9 Cobalt 10.2 0.19 1.9% 103% 809 19.1 2.4% 100% Copper 812 24802 104% 23934 811.03 3.3% Iron 110 2.33 2.1% 104% Lead 105 Magnesium 3261 3365 135 4.0% 103% 558 595 16.0 2.7% 107% Manganese Mercury 53.6 55.0 2.22 4.0% 103% Molybdenum 96.5 92.7 2.71 2.9% 96% Nickel 47.7 49.0 1.74 3.5% 103% Potassium 4293 4279 117.7 2.8% 100% Selenium 105% 65.7 68.8 1.95 2.8% Silver 89.3 90.1 1.81 2.0% 101% 96.17 Sodium 106% 2986 3177 3.0% **Thallium** 100 102 0.943 0.9% 102% Vanadium 37.0 39.1 0.87 2.2% 106% Zinc 758 24.2 92% 821 3.2%