

Metals in Fly Ash – CRM019 Certified Reference Material

SUMMARY

The application note summarizes the digestion of Metals in Fly Ash, CRM019, a Certified Reference Material using ColdBlock™ Digestion CB15S Technology.

Instrument: ColdBlock CB15S sample digester technology, chiller, ICP-OES, ICP-MS

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Digestion Time: 20 Minutes

Acid Used: HNO₃ & HCl

Average ColdBlock Recovery vs. CRM:

- 106% Arsenic
- 97% Manganese
- 100% Lead

METHODOLOGY

1. Chiller temperature was set to -5 °C
2. 0.5g of Fly Ash was weighed and placed into a ColdBlock™ Digestion vessel
3. 12ml HNO₃ was added, and mixed with sample
4. Sample was digested at 80% power for 10 minutes
5. 4mL HCl was added, and sample was digested again at 80% power for 10 minutes

DISCUSSION

- Upon addition of HNO₃, the evolution of reddish brown (NO₂) fumes occurred
- The NO₂ fumes subsided and turned white prior to the addition of HCl
- After 20 minutes the samples are greenish brown, and a minor amount of solid material remains
- Longer digestion times can be used to improve the recoveries of certain elements
- Hydrofluoric acid can be added for a “near total” digestion



CRM019 after 20-minute digestion

Metals in Fly Ash, CRM019 is a Certified Reference Material used for QC verification and/or method development purposes. It was sourced from a wastewater treatment facility in the Western Unit.

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Results

Trace Metals - Fly Ash 3								
Method:	0.5g	12mL HNO ₃ – Digest at 80% for 600 seconds – Add 4mL HCl and digest again for 600 seconds. Bulk to 50mL with 2% HNO ₃						
Element	Certified Value mg/kg (non-certified)	ColdBlock Sample A	ColdBlock Sample B	ColdBlock Sample C	ColdBlock Average (mg/kg)	Stdev	% RSD	% Recovery
Ag	7.35	7.96	8.04	7.3	7.8	0.04	0.5%	106%
As	77.2	80.9	82.1	81.7	82	0.6	0.7%	106%
Ca	(51900)	50962	50182	53225	51456	390	0.8%	99%
Cd	432	414	439	434	429	12.5	2.9%	99%
Co	(26)	24	23	23	23	0.5	2.1%	90%
Cr	55.2	55.1	59.3	54.2	56.2	2.1	3.7%	102%
Cu	279	280	288	284	284	4	1.4%	102%
Fe	(12690)	12473	11135	12581	12063	669	5.5%	95%
Mg	6310	6778	6543	6575	6632	117.5	1.8%	105%
Mn	(480)	456	448	488	464.0	4	0.9%	97%
Na	(50500)	50925	52257	51707	51630	666	1.3%	102%
Ni	22.2	24.3	22.8	25.4	24	0.75	3.1%	109%
Pb	4540	4515	4632	4525	4557	58.5	1.3%	100%
Se	4.11	4.14	4.25	3.7	4.0	0.055	1.4%	98%
V	28.9	29.8	30.2	30.4	30	0.2	0.7%	104%
Zn	22400	21139	21637	21459	21412	249	1.2%	96%