

APPLICATION NOTE

Black Mass

SUMMARY

The application note summarizes the digestion of large sample sizes of active material concentrate, NMC cathode material and shredded lithium-ion batteries using ColdBlock™ Digestion Pro Series Technology.

Instrument: ColdBlock CBL sample digester, chiller, ICP-OES

Published: October 2024

Digestion Time: 25 Minutes

Acid Used: Reverse Aqua Regia

Average ColdBlock Recovery vs. CRM:

- 100% Cobalt
- 100% Lithium
- 99% Nickel

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 20g of each sample was weighed and placed into a ColdBlock™ Digestion vessel
3. 45mL HNO₃ was slowly added (5mL at a time)
4. Wait 5 minutes
5. 10mL of 5%_{v/v} antifoaming solution was added
6. 15mL of HCl was slowly added (3mL at a time)
7. Sample was digested at 60% power for 10 minutes
8. Pause digestion for 5 minutes (optional step: if sample is rising up the tube, add more anti-foaming solution)
9. Digest again at 60% power for an additional 10 minutes
10. Samples were cooled and bulked to 100 mL using 2% HNO₃_{v/v}



Active Material Concentrates after bulk-up to 100mL

DISCUSSION

- Black mass can be very reactive, use caution when adding acid
- If sample continues to rise up the test tube after adding additional anti-foaming solution, consider decreasing the sample size
- After the digestion is complete, samples will most likely be black
- Prior to analysis, samples were allowed to settle overnight. Filtering is also an option.

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Results

Active Material Concentrate A

Analyte	Digestion/ Fusion Results (%)	ColdBlock 1 (%)	ColdBlock 2 (%)	Average	St. Dev	% RSD	% Recovery
Co	2.67	2.73	2.79	2.76	0.0424	1.5	103
Li	7.04	7.29	7.29	7.29	0.0003	0.004	104
Mn	0.003	0.003	0.003	0.003	0.0001	2.3	102
Ni	57	56	57	57	0.30	0.5	99

Active Material Concentrate B

Analyte	Digestion/ Fusion Results (%)	ColdBlock 1 (%)	ColdBlock 2 (%)	Average	St. Dev	% RSD	% Recovery
Co	13.6	13.5	13.1	13.3	0.283	2.1	98
Li	3.60	3.62	3.56	3.59	0.0424	1.2	100
Mn	6.21	6.32	6.35	6.34	0.0212	0.3	102
Ni	11.4	11.6	11.4	11.5	0.14	1.2	101

NMC Cathode

Analyte	Digestion/ Fusion Results (%)	ColdBlock 1 (%)	ColdBlock 2 (%)	Average	St. Dev	% RSD	% Recovery
Co	12.6	12.9	12.4	12.7	0.354	2.8	100
Li	3.43	3.35	3.27	3.31	0.0566	1.7	97
Mn	8.62	8.22	7.99	8.11	0.163	2.0	94
Ni	11.2	10.8	11.1	11.0	0.212	1.9	98

Shredded Lithium-Ion Batteries

Analyte	Digestion/ Fusion Results (%)	ColdBlock 1 (%)	ColdBlock 2 (%)	Average	St. Dev	% RSD	% Recovery
Co	5.97	5.86	5.88	5.87	0.014	0.2	98
Li	6.06	5.96	5.91	5.94	0.035	0.6	98
Mn	4.40	4.30	4.22	4.26	0.057	1.3	97
Ni	40.9	39.9	39.8	39.9	0.071	0.2	97