

# Digestion of Geochemical Soil Reference Materials

## SUMMARY

The application note compares the digestion of Till-2 & Till-3, Geochemical Soil Reference Materials by Natural Resources Canada using ColdBlock™ Digestion CB15S Technology vs traditional EPA and total digestion methods.

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

**Published:** August 2022

**Digestion Time:** 30 Minutes

**Acid Used:** HNO<sub>3</sub> & HCl

**Average ColdBlock Recovery vs. CRM:**

- 92% Till-2 Arsenic
- 102% Till-3 Arsenic
- 103% Till-2 Mercury
- 105% Till-3 Mercury

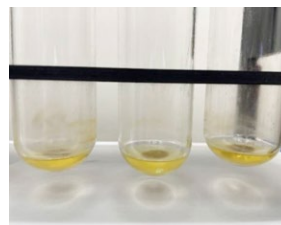
ColdBlock 30-minute digestion shows recoveries well in agreement with a 2–4-hour partial extraction method and/or open vessel digestion method, EPA 3050b, including Mercury and Arsenic. Some silicate bound elements recovered low when compared to “total” multi-acid method that includes Hydrofluoric acid, as expected. Apart from a few analytes, RSD’s <5% demonstrate repeatability across all the replicates.

## METHODOLOGY

1. Chiller temperature was set to -5 °C
2. 0.25g of Till-2 & Till-3 were weighed and placed into ColdBlock™ Digestion vessels
3. 15 mL HNO<sub>3</sub> & 5mL HCl was added and mixed with sample
4. Sample was digested at 70% power for 30 minutes
5. Sample was cooled and bulked to 50mL using 2%HNO<sub>3</sub> + 0.5% HC lv/v

## DISCUSSION

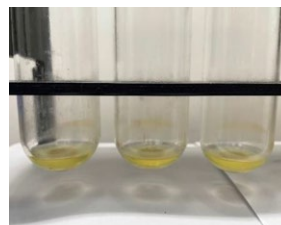
- Upon addition of HNO<sub>3</sub>, the evolution of reddish brown (NO<sub>2</sub>) fumes occurred
- The NO<sub>2</sub> fumes subsided and turned white near the end of the digestion
- After 30 minutes the samples are slightly yellow, and a small amount of solid material remains
- To improve the recovery of Silver, samples can be bulked using 10% HCl v/v
- Longer digestion times can be used to improve the recoveries of certain elements
- Hydrofluoric Acid can be added for a near total digestion



*Till-2 after 30-minute digestion*



*Till-2 after bulk-up*



*Till-3 after 30-minute digestion*



*Till-3 after bulk-up*

*Till-2 was collected in Scissions' Brook, New Brunswick. Till-3 was collected 8 km East of Cobalt, Ontario. These 2 Till samples are characterized for total elements as well as elements that are partially extracted using concentrated HNO<sub>3</sub> & HCl and a digestion time of 2-4 hrs at 95-100°C. In addition, single source data is also provided, derived by EPA 3050 (Open Vessel Acid Digestion) & EPA 3051 (Microwave Assisted Acid Digestion).*

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## Results

Till 2 Results										
Till-2 – ColdBlock Results Values in µg/g unless otherwise noted							% Recoveries of ColdBlock Digestion vs Traditional Methods			
Elements	ColdBlock A	ColdBlock B	ColdBlock C	Average	StDev	% RSD	EPA 3050	EPA 3051	Total	Partial Extraction
Ag	0.24	0.23	0.21	<b>0.23</b>	0.012	5.5%	-	-	-	<b>113%</b>
As	20	20	21	<b>20</b>	0.471	2.3%	-	-	<b>78%</b>	<b>92%</b>
Al	27482	27894	27946	<b>27774</b>	273.172	1.0%	<b>85%</b>	<b>101%</b>	-	-
Ba	98	96	98	<b>97</b>	0.943	1.0%	<b>93%</b>	<b>160%</b>	<b>18%</b>	-
Be	1.3	1.3	1.3	<b>1.3</b>	0.024	1.8%	<b>61%</b>	<b>76%</b>	-	-
Cd	<0.3	<0.3	<0.3	<b>n/a</b>	n/a	n/a	-	-	-	-
Ca	1611	1670	1680	<b>1654</b>	19.442	1.3%	<b>85%</b>	<b>101%</b>	-	-
Cr	34.6	32.2	34.4	<b>33.7</b>	1.090	3.2%	<b>86%</b>	<b>97%</b>	<b>46%</b>	<b>84%</b>
Co	13.3	13.6	14.0	<b>13.6</b>	0.278	2.0%	<b>109%</b>	<b>88%</b>	<b>91%</b>	<b>105%</b>
Cu	142	133	140	<b>138</b>	3.665	2.6%	<b>85%</b>	<b>79%</b>	<b>92%</b>	<b>93%</b>
Fe	33887	33008	33205	<b>33367</b>	376.619	1.1%	<b>98%</b>	<b>86%</b>	<b>87%</b>	<b>104%</b>
Hg (ppb)	78	75	76	<b>76</b>	1.234	1.6%	-	-	-	<b>103%</b>
Pb	271	27.6	27.9	<b>27.5</b>	0.806	3.4%	<b>77%</b>	<b>87%</b>	<b>89%</b>	<b>131%</b>
Mg	7133	6975	7061	<b>7056</b>	64.588	0.9%	<b>93%</b>	<b>83%</b>	-	-
Mn	634	622	630	<b>628</b>	5.129	0.8%	<b>105%</b>	<b>107%</b>	<b>81%</b>	<b>119%</b>
Mo	14.72	14.27	13.1	<b>14.03</b>	0.683	4.9%	<b>75%</b>	-	<b>100%</b>	<b>128%</b>
Ni	24.3	25.9	26.3	<b>25.5</b>	0.760	3.2%	<b>93%</b>	<b>86%</b>	<b>80%</b>	<b>82%</b>
P	634	621	610	<b>622</b>	9.973	1.6%	<b>73%</b>	<b>115%</b>	<b>83%</b>	-
K	3641	3587	3604	<b>3611</b>	22.544	0.6%	<b>83%</b>	<b>107%</b>	-	-
Na	510	574	664	<b>583</b>	63.168	10.8%	<b>111%</b>	<b>129%</b>	-	-
V	40.84	42.09	42.31	<b>41.75</b>	0.647	1.6%	<b>39%</b>	<b>38%</b>	<b>54%</b>	<b>110%</b>
Zn	112.11	110.24	112.79	<b>111.71</b>	1.078	1.0%	<b>101%</b>	<b>99%</b>	<b>86%</b>	<b>96%</b>

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### Results

Till-3 Results										
Till-3 ColdBlock Results Values in µg/g unless otherwise noted							% Recoveries of ColdBlock Digestion vs Traditional Methods			
Elements	ColdBlock A	ColdBlock B	ColdBlock C	Average	StDev	% RSD	EPA 3050	EPA 3051	Total	Partial Extraction
<b>Ag</b>	1.5	1.4	1.3	<b>1.4</b>	0.08	5.8%	-	-	-	<b>88%</b>
<b>As</b>	78	88	90	<b>85</b>	5.1	6.0%	-	-	<b>98%</b>	<b>102%</b>
<b>Al</b>	10629	10424	10658	<b>10570</b>	104.2	1.0%	<b>98%</b>	<b>88%</b>	-	-
<b>Ba</b>	39.8	39.8	39.9	<b>39.8</b>	0.1	0.1%	<b>86%</b>	<b>81%</b>	<b>8%</b>	<b>93%</b>
<b>Cd</b>	<0.2	<0.2	<0.2	-	-	-	-	-	-	-
<b>Ca</b>	5314	5344	5048	<b>5235</b>	133.4	2.5%	<b>84%</b>	<b>92%</b>	-	-
<b>Cr</b>	63.2	60.7	60.3	<b>61.4</b>	1.3	2.1%	<b>92%</b>	<b>95%</b>	<b>50%</b>	<b>84%</b>
<b>Co</b>	10.2	9.7	10.7	<b>10.2</b>	0.4	3.9%	<b>100%</b>	<b>69%</b>	<b>68%</b>	<b>93%</b>
<b>Cu</b>	19.2	19.3	16.8	<b>18.4</b>	1.2	6.4%	<b>105%</b>	<b>112%</b>	<b>84%</b>	<b>80%</b>
<b>Fe</b>	19246	18952	19099	<b>19099</b>	120.0	0.6%	<b>96%</b>	<b>91%</b>	<b>69%</b>	<b>95%</b>
<b>Hg (ppb)</b>	124	103	109	<b>112</b>	8.8	7.9%	-	-	-	<b>105%</b>
<b>Pb</b>	20	23	21	<b>21</b>	1.1	5.4%	<b>89%</b>	<b>92%</b>	<b>82%</b>	<b>133%</b>
<b>Mg</b>	6159	6154	6152	<b>6155</b>	2.9	0.0%	<b>95%</b>	<b>83%</b>	-	-
<b>Mn</b>	317	316	313	<b>315.3</b>	1.7	0.5%	<b>107%</b>	<b>99%</b>	<b>61%</b>	<b>102%</b>
<b>Mo</b>	5	4	4	<b>4.6</b>	0.5	11.1%	<b>91%</b>	<b>76%</b>	<b>228%</b>	-
<b>Ni</b>	26.8	26.7	24.1	<b>25.9</b>	1.2	4.8%	<b>92%</b>	<b>98%</b>	<b>66%</b>	<b>81%</b>
<b>P</b>	426	409	481	<b>439</b>	30.8	7.0%	<b>93%</b>	<b>96%</b>	<b>90%</b>	-
<b>K</b>	1083	1120	1189	<b>1131</b>	44.0	3.9%	<b>93%</b>	<b>117%</b>	-	-
<b>Na</b>	365	367	330	<b>354</b>	17.0	4.8%	<b>105%</b>	<b>83%</b>	-	-
<b>V</b>	44.4	42.5	40.9	<b>42.6</b>	1.4	3.3%	<b>59%</b>	<b>64%</b>	<b>69%</b>	<b>129%</b>
<b>Zn</b>	43.0	40.6	44.4	<b>42.7</b>	1.6	3.7%	<b>100%</b>	<b>100%</b>	<b>76%</b>	<b>99%</b>