

APPLICATION NOTE

OREAS-353 Lead Concentrate

SUMMARY

This application note is for the digestion of OREAS-353, a Lead Concentrate.

Instrument:	ColdBlock CB15S sample digester technology, chiller, ICP-OES, ICP-MS
Published:	August 2022
Digestion Time:	20 Minutes
Acid Used:	Reverse Aqua Regia
Average ColdBlock Recovery vs. CRM:	■ 91% Silver ■ 97% Lead ■ 99% Zinc

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 0.25g of OREAS 353 was weighed and placed into a ColdBlock Digestion vessel
3. 20mL of Reverse Aqua Regia was added (HNO_3 is added first, followed by the HCl) (For elements requiring HF, 20mL Reverse Aqua Regia + 3mL HF was added) (Optional step if using HF: Add 20mL of 4%_{v/v} Boric acid and digest another 10 minutes to re-solubilize any insoluble fluorides precipitating out and neutralize any remaining HF)
4. Sample was digested at 80% power for 20 minutes
5. Sample was cooled and bulked to 50mL using 2% HNO_3 _{v/v}

DISCUSSION

- Upon addition of HNO_3 , the evolution of reddish brown (NO_2) fumes occurred
- After 20 minutes, the samples are dark yellow in color
- Hydrofluoric acid can be added for a "near" total digestion to improve recoveries of certain elements (see table 2 for improved recoveries of Al, Co, Fe, Mg, Mn & Sb)



OREAS 353 before bulk-up
(Inverse Aqua Regia + HF digestion)



OREAS 353 after 20-min Inverse
Aqua Regia digestion

- Boric acid can also be added to re-solubilize any insoluble fluorides and help neutralize any remaining HF)
- For improved silver recoveries bulk up using a solution of $>20\%$ HCl _{v/v}

OREAS 353 is a certified reference material (CRM) prepared from a Pb-Ag concentrate sample sourced from South32 Ltd's Cannington mine plant. The stratabound, metasediment hosted (Broken Hill Type) deposit is located ~200 kms south-east of Mount Isa in north-west Queensland, Australia.

OREAS-353

Lead Concentrate

Results

Table 1: Results of Reverse Aqua Regia digestion

Elements	Expected Value	ColdBlock Value 1	ColdBlock Value 2	Average	% RSD	% Recovery
Ag (ppm)	2184	1987	1969	1978	0.5%	91%
Al (ppm)	2040	794	640	717	10.7%	35%
Ca (ppm)	2360	2411	2291	2351	2.6%	100%
Cd (ppm)	205	198	206	202	2.0%	99%
Co (ppm)	48.8	36.8	50.0	43.4	15.2%	89%
Cu (ppm)	3120	2774	2790	2782	0.3%	89%
Fe (wt.%)	5.35	4.81	4.81	4.81	0.0%	90%
Mg (wt.%)	0.685	0.205	0.206	0.206	0.2%	30%
Mn (wt.%)	0.184	0.147	0.147	0.147	0.0%	80%
Pb (wt.%)	61.26	57.89	60.6	59.25	2.3%	97%
S (wt.%)	15.13	13.66	14.33	14.00	2.4%	92%
Sb (ppm)	1746	606	932	769	21.2%	44%
Zn (wt.%)	4.14	4.12	4.07	4.10	0.6%	99%

Table 2 - Results of adding HF, showing improved recoveries of Al, Co, Fe, Mg, Mn, Sb

Elements	Expected Value	ColdBlock Value 1	ColdBlock Value 2	Average	% RSD	% Recovery
Al (ppm)	2040	2049	1677	1863	0.1%	91%
Co (ppm)	48.8	49.1	51.6	50.4	2.5%	103%
Fe (wt.%)	5.35	5.26	4.87	5.07	0.3%	95%
Mg (wt.%)	0.685	0.681	0.650	0.666	2.3%	97%
Mn (wt.%)	0.184	0.181	0.184	0.183	0.8%	99%
Sb (ppm)	1746	1738	1676	1707	1.8%	98%