Base Metals by Aqua Regia

SUMMARY

This application note summarizes the digestion of base metals by aqua regia in various certified reference materials using ColdBlock™ Digestion Technology.

Instrument:	ColdBlock CB15, chiller, ICP-OES, ICP-MS
Published:	May 2024
Digestion Time:	15 Minutes
Acid Used:	Aqua Regia
Average ColdBlock Recovery Across All CRMs:	■ 99% Copper ■ 102% Nickel ■ 100% Zinc

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 0.5g of each sample were weighed and placed into ColdBlock™ Digestion vessels
3. 16 mL of Aqua Regia (AR) or reverse Aqua Regia (rAR) was added (reverse Aqua Regia was used for high sulphide samples)
4. Samples were digested at 60% power for 15 minutes
5. Samples were cooled and bulked to 50 mL using 15% HCl $_{v/v}$

DISCUSSION

■ For samples with a Sulphide content >10%, it is recommended to invert the ratios of Aqua Regia (3:1 – HNO_3:HCl), add HNO_3 slowly and allow the samples to react before adding HCl (reaction can be vigorous and deep brown/red fumes will be generated (see table 1 for summary of sample types and methods used)

Table 1 – Summary of sample types			
CRM ID	**TYPE**	**Sample Weight**	**Method**
AMIS 0619	Copper Concentrate, Black Mountain, Northern Cape, South Africa	0.5	rAR
AMIS 0566	Copper Concentrate, Philippines	0.5	rAR
GBM318-6	Geochem Base Metal	0.5	AR
OREAS 504b	Porphyry Copper-Gold-Molybdenum	0.5	AR
OREAS 990	Copper-Gold Concentrate	0.5	rAR
OREAS 134b	Zn-Pb-Ag Ore	0.5	rAR

Base Metals by Aqua Regia
Results

AMIS 0619 – Copper Concentrate, Black Mountain, Northern Cape, South Africa

Method:	0.5g	Slowly add 12mL HNO$_3$ and 4mL HCl, digest at 60% power for 15 minutes. Cool, and bulk to 50mL with 15%HCl $_{v/v}$							
Element	Certified Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery
		Low	High							
Co (ppm)	476	438	514	411	408	476	432	31.4	7.3%	91%
Cu (%)	25.14	23.14	27.14	25.5	24.9	25.1	25	0.2	1.0%	100%
Fe (%)	34.22	31.22	37.22	33.7	33.7	34.0	34	0.1	0.4%	99%
Mo (ppm)	1098	975	1221	1040	1030	1050.0	1040	8.2	0.8%	95%
Ni (ppm)	330	294	366	313	306	308	309	2.9	1.0%	94%
Pb (%)	2.72	2.52	2.92	2.59	2.60	2.61	2.60	0.01	0.3%	96%
Zn (%)	1.24	1.17	1.31	1.22	1.25	1.22	1.23	0.01	1.1%	99%

AMIS 0566 – Copper Concentrate, Philippines

Method:	0.5g	Slowly add 12mL HNO$_3$ and 4mL HCl, digest at 60% power for 15 minutes. Cool, and bulk to 50mL with 15%HCl $_{v/v}$							
Element	Certified Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery
		Low	High							
Co (ppm)	48	40	56	42	49	42	44	3.3	7.4%	92%
Cu (%)	21.71	21.11	22.31	21.4	22.4	22.2	22.00	0.4	2.0%	101%
Fe (%)	22.17	21.29	23.05	21.7	22.3	22.2	22.07	0.3	1.2%	100%
Mo (ppm)	87	80	94	84.1	89.1	93.9	89	4.0	4.5%	102%
Pb (ppm)	242	221	263	213	223	208	215	6.2	2.9%	89%
Zn (ppm)	251	228	274	260	273	256	263	7.3	2.8%	105%

GBM318-6 – Geochem Base Metal

Method:	0.5g	Slowly add 4mL HNO$_3$ and 12mL HCl, digest at 60% power for 15 minutes. Cool, and bulk to 50mL with 15%HCl $_{v/v}$							
Element	Certified Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery
		Low	High							
Co (ppm)	49	47.5	50.5	46	47	48	47	0.8	1.7%	96%
Cu (ppm)	2423	2399.4	2446.6	2520	2610	2520	2550	42	1.7%	105%
Ni (ppm)	762	749	775	860	862	869	864	3.9	0.4%	113%
Pb (ppm)	10	9.3	10.7	14	9	10	11	2.3	20.3%	112%
Zn (ppm)	130	122.3	137.7	134	130	131	132	1.7	1.3%	101%

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OREAS 504b – Porphyry Copper-Gold-Molybdenum

Method:	0.5g	Slowly add 4mL HNO$_3$ and 12mL HCl, digest at 60% power for 15 minutes. Cool, and bulk to 50mL with 15% HCl $_{v/v}$							
Element	Certified Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery
		Low	High							
Co (ppm)	18.7	18.1	19.4	19	19	19	18.9	0.05	0.2%	101%
Cu (%)	1.1	1.09	1.11	1.06	1.05	1.06	1.1	0.005	0.4%	96%
Fe (%)	6.71	6.51	6.91	6.57	6.63	6.58	6.59	0.03	0.4%	98%
Ni (ppm)	30	29.1	31	29	30	30	30	0.2	0.6%	99%
Mo (ppm)	476	467	485	459	457	459	458	0.9	0.2%	96%
Pb (ppm)	26.2	25.1	27.3	25.2	28.2	24.8	26.1	1.5	5.8%	99%
Zn (ppm)	96	94	99	86	90	82	86	3.3	3.8%	90%

OREAS 990 – Copper-Gold Concentrate

Method:	0.5g	Slowly add 12mL HNO$_3$ and 4mL HCl, digest at 60% power for 15 minutes. Cool, and bulk to 50mL with 15% HCl $_{v/v}$							
Element	Certified Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery
		Low	High							
Cu (%)	16.99	16.72	17.25	16.6	16.6	15.5	16.21	0.5	3.2%	95%
Fe (%)	20.07	19.38	20.77	19.0	18.7	18.9	18.87	0.1	0.7%	94%
Ni (ppm)	93	87	100	96	94	96	95	0.9	1.0%	103%
Mo (ppm)	116	108	125	116	116	120	117	1.9	1.6%	101%
Pb (%)	8.62	8.51	8.73	8.28	8.07	8.61	8.32	0.2	2.7%	97%
Zn (%)	13.43	13.1	13.75	13.69	13.48	13.49	13.55	0.1	0.7%	101%

OREAS 134b – Zn-Pb-Ag Ore

Method:	0.5g	Slowly add 12mL HNO$_3$ and 4mL HCl, digest at 60% power for 15 minutes. Cool, and bulk to 50mL with 15% HCl $_{v/v}$							
Element	Certified Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery
		Low	High							
Co (ppm)	104	92	116	95	96	94	95	0.8	0.9%	91%
Cu (ppm)	1337	1284	1391	1290	1300	1300	1297	4.7	0.4%	97%
Fe (%)	12.25	11.71	12.8	11.7	11.6	11.8	11.7	0.08	0.7%	96%
Pb (%)	13.31	12.85	13.78	13.1	12.9	13.1	13.03	0.1	0.7%	98%
Zn (%)	17.7	17.04	18.36	17.7	17.9	18.1	17.9	0.2	0.9%	101%

info@coldblock.ca
coldblock.ca/contact-us