

OREAS 503c – Porphyry Copper-Gold

SUMMARY

The application note summarizes the digestion of OREAS 503c, a Porphyry Copper-Gold Certified Reference Material using ColdBlock™ Digestion Pro Series Technology.

Instrument: ColdBlock CBM & CBL sample digesters, chiller, HF compatible liners, ICP-MS & ICP-OES

Published: February 2023

Digestion Time: 20 Minutes (Aqua Regia) & 30 Minutes (Strong Acid)

Acid Used: Aqua Regia & Aqua Regia, HF & H₃BO₃

Average ColdBlock Recovery vs. CRM:

- 101% Gold
- 99% Arsenic
- 101% Copper

METHODOLOGY

Aqua Regia Digestion (GOLD)

1. Chiller temperature was set to -5°C
2. 30g was weighed and placed into a ColdBlock™ Digestion vessel
3. 120 mL of Aqua Regia was added (HNO₃ was added first, followed by HCl)
4. Sample was digested at 80% power for 20 minutes
5. Samples were cooled and bulked to 200mL using 20% HCl_{v/v}

Strong Acid Digestion

1. Chiller temperature was set to -5°C
2. 0.25g was weighed and placed into a ColdBlock™ Digestion vessel
3. 20 mL of Aqua Regia was added
4. Sample was digested at 80% power for 20 minutes
5. 20mL of 10%_{v/v} Boric acid was added
6. Samples were digested again at 80% power for 10 minutes
7. Samples were cooled and bulked to 50mL using 2% HNO₃ + 0.5% HCl_{v/v}

DISCUSSION

Aqua Regia Digestion

- If the Sulfide content of your sample is > 10 wt.% - reverse the ratios of Aqua Regia and use 1:3, HCl:HNO₃
- Always add HNO₃ first, slowly, in small increments. (Reddish brown NO₂ fumes might form) If a reaction occurs wait until it settles before adding the HCl.
- ICP-MS was used for analysis of Gold - see Table 1 for results

Strong Acid Digestion

- The addition of Boric acid will help re-solubilize any insoluble fluorides and will help neutralize any remaining HF in solution
- If Silver precipitates out of solution as AgCl, bulk up with >10% HCl_{v/v}
- If the Sulfide content of your sample is > 10 wt.% - reverse the ratios of Aqua Regia and use 1:3, HCl:HNO₃ - always add the Nitric acid first (reddish brown NO₂ fumes might form)
- See table 2 for results.

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Figure 1 - After Bulk-up using Strong Acid Digestion



Figure 2 - After digestion using Aqua Regia digestion

OREAS 503c was prepared from a blend of porphyry copper-gold ore, barren granodiorite, and a minor quantity of Cu-Mo concentrate. The ore was sourced from the Ridgeway underground mine located in the Cadia Valley Operations (CVO) situated in central western New South Wales, Australia. ore.com.au

OREAS 503C; Porphyry Copper-Gold-Molybdenum; COA-1313-OREAS 503c_2; Ore research & Exploration; Melbourne, Australia (June,2018)

Results

Table 1 - OREAS 503c by 30g Aqua Regia Digestion

OREAS 503c - Porphyry Copper-Gold										
Method:	30g	120mL Aqua Regia, digested 80% for 20 minutes								
Element	Certified Fire Assay Value	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery vs certified Fire Assay
		Low	High							
Au	0.698	0.693	0.703	0.697	0.694	0.716	0.702	0.04	4.1%	101%

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Table 2 - OREAS 503c by Strong Acid Digestion

OREAS 503c - Porphyry Copper Gold										
Method:	0.25g	20mL Aqua Regia + 3 mL HF digested at 80% for 20 minutes, add 20mL of 10% Boric Acid - and digest again at 80% for another 10 mins								
Element	OREAS Certified 4-acid Value (ppm)	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery vs certified 4-Acid value
		Low	High							
Ag	0.83	0.788	0.871	0.87	0.82	0.83	0.84	0.04	4.1%	101%
Al	73700	72400	75000	71285	71285	72526	71698	585.10	0.8%	97%
As	34.5	33.8	35.2	35.1	33.2	34.1	34.1	0.76	2.2%	99%
Ba	985	965	1004	1093	1095	1221	1137	59.91	5.3%	115%
Be	2.65	2.59	2.71	2.63	3.54	2.72	2.96	0.46	14.7%	112%
Bi	0.6	0.58	0.62	0.6	0.7	0.6	0.6	0.07	10.6%	104%
Ca	26300	25800	26900	26685	26712	27314	26904	290.26	1.1%	102%
Cd	0.22	0.19	0.25	0.22	0.22	0.2	0.21	0.01	4.4%	97%
Ce	63	61	64	61	61	63	62	0.71	1.2%	98%
Co	14.6	14.4	14.8	14.3	14.6	13.6	14.2	0.42	3.0%	97%
Cr	68	66	70	74	71	71	72	1.36	1.9%	106%
Cs	10.3	10.1	10.4	10.9	11.1	11.2	11.1	0.13	1.1%	107%
Cu	5380	5330	5430	5200	5223	5309	5244	46.63	0.9%	97%
Dy	4.21	3.97	4.46	3.99	3.94	4.25	4.06	0.14	3.3%	96%
Er	2.37	2.31	2.43	2.38	2.37	2.46	2.40	0.04	1.7%	101%
Eu	1.26	1.18	1.34	1.21	1.26	1.14	1.20	0.05	4.1%	96%
Fe	51700	50800	52500	50408	50680	50284	50457	165.42	0.3%	98%
Ga	18.5	18.3	18.8	17.9	18.1	17.3	17.7	0.32	1.8%	96%
Gd	4.75	4.48	5.01	4.69	4.8	4.89	4.79	0.08	1.7%	101%
Hf	2.4	2.36	2.44	2.5	2.1	2.3	2.3	0.20	8.7%	96%
Ho	0.85	0.84	0.86	0.8	0.83	0.82	0.82	0.01	1.5%	96%
K	31800	31300	32300	31156	31085	31041	31094	47.19	0.2%	98%
La	32	31.3	32.7	33	31	32	32	0.79	2.5%	100%
Li	31.1	30.5	31.8	31.3	31.4	31.6	31.5	0.14	0.4%	101%
Lu	0.34	0.32	0.35	0.32	0.32	0.34	0.33	0.01	2.9%	96%
Mg	15000	14800	15300	15275	15286	15284	15282	4.54	0.0%	102%
Mn	540	530	540	560	546	553	553	5.65	1.0%	102%
Mo	318	313	322	321	328	326	325	2.90	0.9%	102%
Na	19900	19700	20200	17338	16982	17027	17116	158.43	0.9%	86%
Nb	16.6	16.1	17	17.7	17.9	18.8	18.1	0.48	2.6%	109%

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Table 2 - OREAS 503c by Strong Acid Digestion

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Method:	0.25g	20mL Aqua Regia + 3 mL HF digested at 80% for 20 minutes, add 20mL of 10% Boric Acid - and digest again at 80% for another 10 mins								
Element	OREAS Certified 4-acid Value (ppm)	95% Confidence Limits		Sample A	Sample B	Sample C	Average (ppm)	Stdev	% RSD	% Recovery vs certified 4-Acid value
		Low	High							
Nd	27.7	26.2	29.3	26.9	25.2	26.7	26.3	0.76	2.9%	95%
Ni	38.6	37.6	39.5	45.6	37.3	39.9	40.9	3.44	8.4%	106%
Pb	20.6	20.2	21.1	20.3	19.7	20.3	20.1	0.28	1.4%	98%
Pr	7.26	7.07	7.45	7.20	6.79	7.10	7.03	0.18	2.5%	97%
Rb	178	175	180	174	170	171	172	1.70	1.0%	96%
S	5860	5750	5960	5840	5820	5814	5824	11.21	0.2%	99%
Sb	3.6	3.53	3.67	3.9	3.9	4.0	3.9	0.01	0.2%	109%
Sc	13.2	12.8	13.7	13.7	14.3	13.5	13.8	0.30	2.1%	105%
Se	3.31	3.09	3.53	3.02	3.04	3.05	3.04	0.01	0.4%	92%
Sm	5.26	5	5.52	5.15	5.12	5.39	5.22	0.12	2.3%	99%
Sr	336	331	340	327	322	322	324	2.36	0.7%	96%
Tb	0.73	0.72	0.75	0.69	0.74	0.71	0.71	0.02	2.9%	98%
Te	0.35	0.33	0.37	0.47	0.48	0.38	0.44	0.05	10.1%	127%
Th	16.5	16	17.1	15.7	16.2	16.6	16.2	0.37	2.3%	98%
Ti	4530	4470	4590	4751	4832	4181	4588	289.61	6.3%	101%
Tl	0.86	0.84	0.89	0.89	0.99	0.89	0.92	0.05	5.1%	107%
Tm	0.33	0.32	0.35	0.32	0.32	0.34	0.33	0.01	2.9%	99%
U	4.47	4.32	4.61	4.58	4.89	5.33	4.93	0.31	6.2%	110%
V	123	121	126	124	124	126	125	0.71	0.6%	101%
Y	23.3	22.9	23.7	22.0	21.0	21.2	21.4	0.42	2.0%	92%
Yb	2.24	2.1	2.38	2.15	2.16	2.30	2.20	0.07	3.1%	98%
Zn	87	85	88	91	89	N/A	90	1.05	1.2%	104%
Zr	75	72	78	68	63	60	64	3.26	5.1%	85%