ColdBlock™ Technologies Inc.

APPLICATION NOTE

Gold by Aqua Regia (large sample size)

SUMMARY

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

Instrument:	ColdBlock CBL, chiller, ICP-MS
Published:	May 2024
Digestion Time:	15 Minutes
Acid Used:	Aqua Regia
Average ColdBlock Recovery vs. CRM:	■ 101% Gold

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 30g of each sample were weighed and placed into ColdBlock™ Digestion vessels (15g for high sulphide samples)
3. 120 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 100% power for 15 minutes
5. Samples were cooled and bulked to 200 mL using 15% HCl $_{v/v}$

DISCUSSION

- For samples with a Sulphide content >10%, it is recommended to drop the sample size to 15g and invert the ratios of Aqua Regia (3:1 – HNO_3:HCl), add HNO3 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
OREAS 991	Copper-Gold Concentrate	15	rAR
OREAS 990	Copper-Gold Concentrate	15	rAR
OREAS 602	High Sulphidation Epithermal Ag-Cu-Au Ore	30	AR
OREAS 251	Gold Oxide Ore	30	AR
OREAS 504b	Porphyry Copper-Gold-Molybdenum	30	AR
OREAS 992	Copper Sulphide	15	rAR
OREAS 604	High Sulphidation Epithermal Ag-Cu-Au Ore	30	AR
OREAS 605	High Sulphidation Epithermal Ag-Cu-Au Ore	30	AR
OREAS 501C	Porphyry Copper-Cold Molybdenum	30	AR
OREAS 520	Iron Oxide Copper-Gold Ore	30	AR
OREAS 221	Gold Ore (Andy Well Gold Mine, Western Australia	30	AR
OREAS 601	High Sulphidation Epithermal Ag-Cu-Au Ore	30	AR
OREAS 905	Copper-Gold Oxide Ore	30	AR
OREAS 620	VHMS Zn-Pb-Cu-Ag-Au Ore	30	AR

Gold by Aqua Regia (large sample size) Results

OREAS 991 – Copper-Gold Concentrate										
Method:	**15g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	47.04	46.70	47.37	44.27	45.73	46.8	45.60	1.04	2.3%	97%

OREAS 990 – Copper-Gold Concentrate										
Method:	**15g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	76.11	75.65	76.57	79.07	82.49	81.47	50.83	0.59	1.2%	108%

OREAS 602 – High Sulphidation Epithermal Ag-Cu-Au Ore										
Method:	**30g**	**Slowly add 90mL HCl, and 30mL HNO_3 and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	1.95	1.93	1.98	1.92	1.97	1.96	81.01	1.43	1.8%	106%

OREAS 251 – Gold Oxide Ore										
Method:	**30g**	**Slowly add 90mL HCl, and 30mL HNO_3 and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	0.504	0.498	0.510	0.522	0.542	0.504	1.950	0.02	1.1%	100%

OREAS 504b – Porphyry Copper-Gold-Molybdenum										
Method:	**30g**	**Slowly add 90mL HCl, and 30mL HNO_3 and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	1.61	1.59	1.62	1.59	1.56	1.55	1.57	0.02	1.1%	97%

Continued...

Gold by Aqua Regia (large sample size) Results

OREAS 992 – Copper Sulphide										
Method:	**15g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Indicative Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	7.92	N/A	N/A	7.91	7.46	7.79	7.72	0.19	2.5%	97%

OREAS 604 – High Sulphidation Epithermal Ag-Cu-Au Ore										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	1.43	1.41	1.45	1.41	1.48	1.47	1.45	0.03	2.1%	102%

OREAS 605 – High Sulphidation Epithermal Ag-Cu-Au Ore										
Method:	**15g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	1.67	1.63	1.70	1.65	1.69	1.68	1.67	0.02	1.0%	100%

OREAS 501c – Porphyry Copper-Cold Molybdenum										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	0.221	0.219	0.224	0.22	0.233	0.22	0.224	0.01	2.7%	102%

OREAS 520 – Iron Oxide Copper-Gold Ore										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	0.176	0.174	0.178	0.167	0.166	0.166	0.166	0.0003	0.2%	94%

Continued...

Gold by Aqua Regia (large sample size)

OREAS 221 – Gold Ore (Andy Well Gold Mine, Western Australia)										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	1.06	1.05	1.07	1.04	1.05	1.07	1.05	0.01	1.2%	99%

OREAS 601 – High Sulphidation Epithermal Ag-Cu-Au Ore										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	0.780	0.769	0.791	0.793	0.787	0.813	0.80	0.01	1.4%	102%

OREAS 905 – Copper-Gold Oxide Ore										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	0.391	0.388	0.394	0.407	0.393	0.4	0.40	0.01	1.4%	102%

OREAS 620 – VHMS Zn-Pb-Cu-Ag-Au Ore										
Method:	**30g**	**Slowly add 90mL HNO_3 (5mL at a time) once sample has finished reacting, add 30mL HCl and digest at 100% for 15 minutes. Let cool, and bulk to 200mL with 15% $_{v/v}$ HCl**								
Element	**Certified Fire Assay (ppm)**	**95% Confidence Limits**		**Sample A**	**Sample B**	**Sample C**	**Average (ppm)**	**Stdev**	**% RSD**	**% Recovery**
		Low	**High**							
Au	0.685	0.676	0.693	0.687	0.7	0.693	0.693	0.01	0.8%	101%

ColdBlock™ Technologies Inc.

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