A NEW, VERSATILE TOOL FOR SAMPLE PREPARATION: COLDBLOCK

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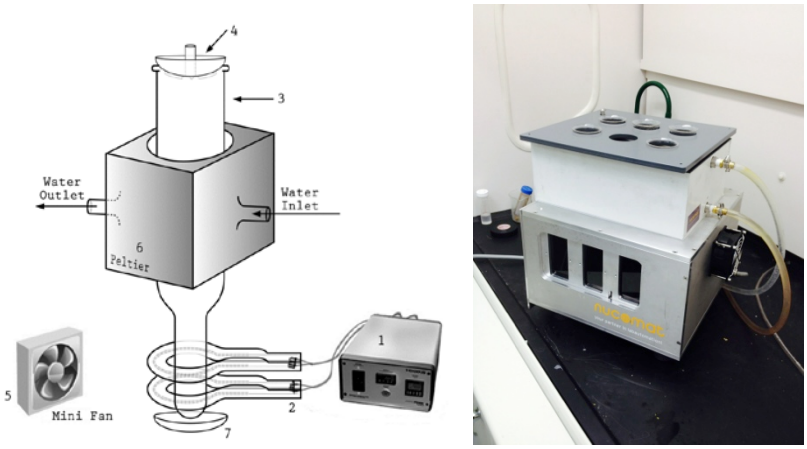
Introduction

ColdBlock is a new technology for ore and mineral digestion that provides rapid (typically less than 15 minutes) sample digestions, delivers excellent recoveries of elements, and avoids the use of hazardous reagents such as perchloric acid. Data for a wide range of sample digestions will be presented that illustrate the versatility of this technology for samples ranging from base metal ores, sulfidic copper concentrate, gold- and silver-containing ores, to refractory chromites. Analysts have noted that recoveries of gold are often poor by fire assay and acid digestion methods (Hall, 1989). The results of an investigation of a modified aqua regia method for the determination of gold and silver show excellent recoveries. In addition, a practical solution to the problems of wash-in and wash-out of gold that are routinely encountered in silica-based sample introduction systems in ICP-based instruments will be discussed.



A **B**

Figure 1: A - Schematic of ColdBlock[1] and B - commercial prototype

[1] Figure reproduced courtesy of the Royal Society of Chemistry

Methodology

Contaminated Soil – the first test of ColdBlock

0.25 gram sample, 7 mL of combined leaching mixture (*aqua regia* and H_2O_2); 15 minute digestion (Wang 2014a)[2]

Results[3]

Table 1: Contaminated Soil SRM (SCP Science SS-1)

Element	Measured (mg kg^{-1})	Certified (mg kg^{-1})	Recovery (%)	RSD (%)
Al	11400±200	12163±409	94	1.9
Ca	50100±400	50265±1213	100	0.9
Cr	90±2	103±6.1	88	2.4
Cu	410±8	403±10	102	2.8
Fe	75600±900	72000±2273	105	1.7
Mg	9500±100	9690±230	98	1.1
Mn	780±10	737±19	106	2.1
Ni	64±1	59.2±1.3	108	1.8
Pb	830±20	764±15	108	2.7
V	27.0±1.0	27.2±1.4	101	3.3
Zn	1200±12	1114±37	107	1.5

Methodology

Chromite Determination

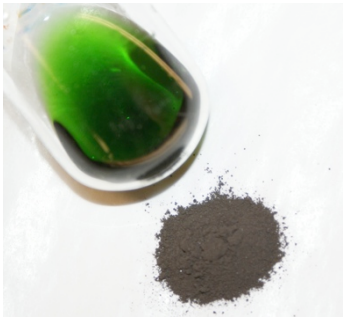


Figure 2. Chromite sample (below) and digested sample

Chromite samples are commonly prepared by fusion. Where acid digestion is available, laboratories caution that such digestions are difficult and time-consuming. CEM proposes a method for the microwave digestion of chromite, using a combination of sulphuric, phosphoric, and nitric acid in a two-step process (including opening the microwave vessel after cooling before the second step) that achieves digestion with 600 psi pressure and a total handling time that exceeds 2 hours (CEM, 1999). The ColdBlock technique achieves complete recovery of chromium with a 12-minute digestion at atmospheric pressure.

[2] These data were produced using a single-ring ColdBlock prototype.
[3] These data are reproduced courtesy of the Royal Society of Chemistry

Results

Table 2. Trace elements in copper concentrate CCU-1d using a 10-12-minute ColdBlock digestion using modified *aqua regia*

CCU-1D	Certified values (µg g^{-1})	ColdBlock™ (HNO_3/HBr, n=3)		INAA (n=2)	
		Measurement (µg g^{-1})	Recovery (%)	Measurement (µg g^{-1})	Recovery (%)
Au (µg g^{-1})	14.01±0.18	13.2±0.3	94.0	12.1±0.8	86.4
Cu (%)	23.99±0.04	24.3±0.5	101.2	-	-
Fe (%)	29.3±0.4	29.9±0.3	102.4	29.8±0.5	101.7
Pb (%)	0.262±0.005	0.267±0.008	102.0	-	-
Zn (%)	2.63±0.04	2.69±0.08	102.1	2.67±0.08	101.5

Methodology

Elimination of gold memory effect

In determinations of gold, a 0.5% L-cysteine solution (in 1.0% HCl matrix) eliminates the memory effect (reduces wash-in and wash-out times substantially), that with nitric or hydrochloric acid can take up to 10 minutes. (Wang, 2014b).

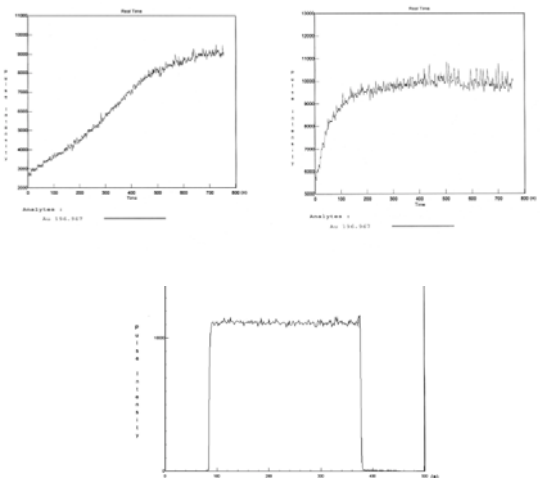


Figure 3. Gold memory effect – top left - nitric acid; top right - hydrochloric acid, and – bottom - elimination by L-cysteine[4]

[4] Figures reproduced courtesy of the Royal Society of Chemistry

References

CEM,1999**.** Chromite Ore: Microwave Sample Preparation Note: 5OS-47 http://www.uwm.edu.pl/kchemsr/MARS/OXIDSUL/Chromit.pdf Accessed February 4, 2015.

HALL, G.E.M., VAIVE, J.E., COOPE, J.A. & WEILAND, E.F. 1989. Bias in the analysis of geological materials for gold using current methods. Journal of *Geochemical Exploration*, **34**, 157-171.

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