Certificate of Analysis

First issued: August 2013

Version: April 2023

HV-2a

Certified Reference Material for Copper-Molybdenum Ore

Table 1 – HV-2a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	1.448	0.089	0.089	0.048
Al (no AD2) [a]	%	7.96	0.10	0.20	0.11
As	µg/g	12.1	0.8	1.7	1.2
Ba (no AD2) [a]	µg/g	869	15	29	15
Ca (no AD2) [a]	%	1.891	0.035	0.080	0.037
Ce [b]	µg/g	19.1	0.6	1.3	0.8
Co	µg/g	3.40	0.10	0.44	0.27
Cr	µg/g	100	4	11	8
Cs (no AD2) [a]	µg/g	2.70	0.09	0.16	0.10
Cu	%	0.3808	0.0057	0.0076	0.0032
Dy [c]	µg/g	1.126	0.053	0.085	0.063
Er [c]	µg/g	0.646	0.039	0.055	0.039
Fe (no AD2) [a]	%	2.044	0.036	0.095	0.041
Ga (no AD2) [a]	µg/g	19.56	0.45	0.80	0.48
Gd [b]	µg/g	1.40	0.09	0.11	0.08
K (no AD2) [a]	%	2.31	0.03	0.10	0.06
La	µg/g	9.1	0.5	1.4	0.7
Mg (no AD2) [a]	%	0.329	0.008	0.030	0.015
Mn [b]	µg/g	545	12	29	15
Mo	µg/g	125.4	2.5	6.2	3.1

cont'd

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Table 1 – HV-2a Certified Values *cont'd*

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Na [c]	%	2.335	0.036	0.066	0.037
Nd [b, e]	µg/g	8.77	0.40	0.57	0.43
Ni	µg/g	6.47	0.32	0.55	0.33
P	%	0.0427	0.0025	0.0038	0.0023
Pb	µg/g	6.9	0.5	1.2	0.6
Rb (no AD2)[a]	µg/g	48.3	1.0	2.8	1.7
S	%	0.344	0.008	0.034	0.013
Sb (no AD2) [a]	µg/g	0.689	0.058	0.085	0.064
Si [d]	%	31.34	0.23	0.59	0.40
Sm [c]	µg/g	1.69	0.13	0.13	0.09
Sr (no AD2) [a]	µg/g	472	9	19	9
Th	µg/g	1.28	0.09	0.13	0.07
Ti (no AD2) [a]	%	0.128	0.004	0.017	0.010
U	µg/g	1.08	0.09	0.12	0.07
Zn (no AD2) [a]	µg/g	56.5	2.3	3.9	2.2

a set(s) using digestion by two acids (hydrochloric and nitric) was/were excluded as method outliers based on statistical tests

b sets using microwave digestion by three acids (hydrochloric, nitric and hydrofluoric acids) were excluded as method outliers based on statistical tests

c methods using only digestion by four acids (hydrochloric, nitric, hydrofluoric and perchloric), microwave digestion with 3 acids and various fusions were used by the laboratories

d all accepted sets of data except one were derived from various fusions

e methods using only digestion by four acids and various fusions were included based on statistical tests

Table 2 – HV-2a Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Be (no AD2) [a, b]	µg/g	1.02	0.04	0.13	0.14
Bi	µg/g	1.58	0.11	0.19	0.15
Eu [c]	µg/g	0.502	0.029	0.046	0.037
loss on ignition [d]	%	3.01	0.05	0.29	0.21
Lu (AD4, FUS) [e]	µg/g	0.109	0.008	0.019	0.015
Pr [c]	µg/g	2.32	0.077	0.083	0.075
V (no AD2) [b]	µg/g	52.2	1.2	4.7	2.5
W (no AD2) [b]	µg/g	7.89	0.52	0.52	0.46
Y	µg/g	5.96	0.13	0.81	0.45
Yb [f, g]	µg/g	0.680	0.047	0.069	0.048
Zr (FUS) [a, h]	µg/g	65.8	5.0	5.0	4.9

a statistical analysis of the data warrants classification as provisional despite only 6 sets of data
b sets using digestion by two acids (hydrochloric and nitric) were excluded as method outliers based on statistical tests
c sets other than digestion using four acids (hydrochloric, nitric, hydrofluoric and perchloric) and various fusions were excluded as method outliers based on statistical tests
d samples of 1 to 10 grams were ignited for 0.5 to 2 hours at 950 to 1050°C
e methods using digestion by four acids and various fusions were used by the laboratories
f methods using digestion by four acids, microwave digestion with 3 acids (hydrochloric, nitric and hydrofluoric acids) and various fusions were used by the laboratories
g data fulfilled the conditions for certified but the element was reclassified as provisional since a considerable amount of the data has only one significant figure
h only various fusion methods were used by the laboratories

Table 3 – HV-2a Informational Values (semi-quantitative only)

Analyte	Units	Mean	No. accepted laboratories / values	Analyte	Units	Mean	No. accepted laboratories / values
C	%	0.4	3 / 15	Sc	µg/g	3	7 / 35
Cd	µg/g	0.2	5 / 25	Se	µg/g	0.7	4 / 20
Hf (FUS) [a]	µg/g	2	4 / 20	Sn	µg/g	1.2	6 / 30
Ho (FUS) [a]	µg/g	0.2	5 / 25	Ta [f]	µg/g	0.2	6 / 30
Fe (AD2) [b]	%	1.6	3 / 15	Tb [g]	µg/g	0.19	5 / 25
Li (no AD2) [c]	µg/g	11	5 / 25	Tl	µg/g	0.1	4 / 20
moisture [d]	%	0.3	6 / 28	Tm [a]	µg/g	0.10	5 / 25
Nb	µg/g	2	7 / 35	Zn (AD2) [b]	µg/g	40	3 / 15
Re [e]	µg/g	0.1	3 / 15				*Cont'd*

a   only various fusion methods were used by the laboratories
b   methods using only digestion by two acids (hydrochloric and nitric) were used by the laboratories
c   the accepted sets included digestion by four acids (hydrochloric, nitric, hydrofluoric and perchloric) and microwave digestion with 3 acids (hydrochloric, nitric and hydrofluoric)
d   samples of 1-10 grams were dried for 0.5 to 12 hours at 100 to 105°C
e   methods using only acid digestion were used by the laboratories
f   methods using digestion by four acids and fusion with lithium metaborate were used by the laboratories
g   methods using digestion by four acids and various fusions were used by the laboratories

SOURCE
HV-2a is a copper-molybdenum ore donated by Teck Highland Valley Copper Partnership in British Columbia, Canada. This is the same source as its predecessors, HV-2 and HV-1.

DESCRIPTION
The mineral species include: quartz (38.0%), albite (35.2%), muscovite (17.5%), calcite (2.1%), epidote (1.5%), kaolinite (1.3%), various other silicates in minor amounts (1.2%), magnetite (1.0%), chalcopyrite (0.5%), bornite (0.4%), ankerite and apatite (each at 0.3%), diopside, siderite and titanite (each at 0.2%), pyrite (0.1%) and hypersthene (0.03%).

INTENDED USE
HV-2a is suitable for the analysis of copper and molybdenum and various other elements in ores in concentrations ranging from major, minor to trace levels. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE
HV-2a should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. CanmetMINING is not responsible for changes occurring after shipment.

HANDLING INSTRUCTIONS
Normal safety precautions for handling fine particulate matter are suggested, such as the use of safety glasses, breathing protection, gloves and a laboratory coat.

METHOD OF PREPARATION

The raw material was ground and sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction was 73%. The product was blended, and then bottled in 200-gram units. This is the only size that is available.

HOMOGENEITY

The homogeneity of the stock was investigated using fifteen bottles chosen according to a stratified random sampling scheme. Three subsamples were analyzed from each bottle. Subsamples of 0.25g grams were digested using hydrofluoric, hydrochloric, nitric and perchloric acids, and analyzed by inductively coupled plasma – optical emission spectrometry for copper, and inductively coupled plasma – mass spectrometry for molybdenum. Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters.

A one–way analysis of variance technique (ANOVA)[1] was used to assess the homogeneity of these elements. No significant between-bottle variation was observed for copper and molybdenum.

CERTIFIED VALUES

Twenty-eight industrial, commercial and government laboratories participated in an interlaboratory measurement program using methods of their own choice.

Methods for the determination of the elements included digestion with various combinations of acids on a hot plate or in a microwave oven and various types of fusions followed by the determination using atomic absorption spectrometry, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry. X-ray fluorescence on a pressed pellet or fused bead was also used for many elements.

The concentration of carbon was determined using combustion followed by infrared spectrometry. The concentration of sulphur was determined using various acid digestions and fusions followed by inductively coupled plasma – optical emission spectroscopy, inductively coupled plasma – mass spectrometry, and X-ray fluorescence. Additionally, combustion followed by infrared spectrometry and gravimetric analysis were used.

ANOVA was used to calculate the consensus values and other statistical parameters from the interlaboratory measurement program. Values are deemed to be certified if derived from 10 or more sets of data that meet CCRMP's statistical criterion regarding the agreement of the results. Thirty-five parameters were certified (see Table 1). Some certified elements exclude digestion using two acids based on statistical tests.

Full details of all work, including the statistical analyses, the methods and the names of the participating laboratories are contained in the Certification Report. For more details on how to use reference material data to assess laboratory results, users are directed to ISO Guide 33:2000, pages 14-17, and the publication, "Assessment of laboratory proficiency using CCRMP reference materials", at www.ccrmp.ca.

UNCERTIFIED VALUES

Eleven provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CCRMP statistical criterion regarding agreement; or 10 or more sets of data, that do not fulfill the CCRMP statistical criteria required for certification; or 6 or 7 sets of data for which the statistical analysis of the data warranted provisional status. This latter group includes beryllium by all methods except digestion using two acids; and zirconium by various fusion methods. Informational values for 17 parameters, shown in Table 3, were derived from the means of a minimum of 3 sets of data.

TRACEABILITY
The values quoted herein are based on the consensus values derived from the statistical analysis of the data from the interlaboratory measurement program, and the standards used by the individual laboratories. The report gives the available details.

CERTIFICATION HISTORY
HV-2a was released as a new material in August 2013. In April 2023 the certificate was re-issued with the units for titanium changed from µg/g to %.
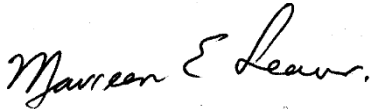
PERIOD OF VALIDITY
The certified values are valid until August 31, 2034.

LEGAL NOTICE
CanmetMINING has prepared this reference material and statistically evaluated the analytical data of the interlaboratory measurement program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies CanmetMINING from and against all liability and costs arising out of the use of this material and information.

CERTIFYING OFFICERS

_____   _____
Maureen E. Leaver – CCRMP Coordinator   Joseph Salley – Data Processor

FOR FURTHER INFORMATION

The Certification Report is available free of charge upon request to:
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REFERENCES
1. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960.