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CCRMP
Canadian Certified Reference Materials Project

PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: November 2012 Version: November 2012

MP-2a

Certified Reference Material for Tungsten-Molydenum Ore

Table 1 – MP-2a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	4.82	0.16	0.45	0.21
Al no AD [a]	%	5.99	0.09	0.18	0.11
Ba	µg/g	12.3	1.1	1.6	1.0
Be no AD2 [b]	µg/g	1.25	0.04	0.15	0.11
Bi	µg/g	989	21	56	23
Ca	%	3.22	0.07	0.24	0.10
Cd	µg/g	14.5	0.4	1.5	0.7
Ce [c]	µg/g	357	7	16	9
Co	µg/g	5.50	0.25	0.37	0.18
Cr	µg/g	150	6	18	8
Cs [c]	µg/g	5.78	0.15	0.25	0.17
Cu	µg/g	459	9	24	9
Dy [d]	µg/g	32.5	0.7	1.8	1.0
Fe	%	5.00	0.08	0.22	0.08
Gd [d]	µg/g	24.8	0.5	1.8	0.9
Hf [c]	µg/g	9.40	0.25	0.44	0.29
La [b]	µg/g	157	4	10	5
Li [e]	µg/g	81	3	11	7
Lu [c]	µg/g	4.36	0.12	0.31	0.17
Mg	µg/g	923	41	77	37

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

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Mn	%	0.1018	0.0024	0.0067	0.0028
Mo	%	0.1586	0.0033	0.0075	0.0031
Nb [f]	µg/g	97	3	10	6
Nd [c]	µg/g	117.9	2.5	5.7	2.8
Pb	%	0.277	0.004	0.016	0.006
Pr [d]	µg/g	38.5	0.9	2.7	1.4
Rb [g]	µg/g	229	5	17	9
S	%	0.716	0.022	0.049	0.022
Sc [h]	µg/g	4.87	0.26	0.53	0.37
Si [i]	%	31.2	0.4	1.2	0.7
Sm [c]	µg/g	26.7	0.6	1.5	0.9
Sn [i]	µg/g	537	11	24	18
Sr	µg/g	12.3	0.4	1.0	0.5
Ta [j]	µg/g	11.6	0.4	1.5	0.9
Tb [c]	µg/g	4.82	0.10	0.34	0.17
Th [c]	µg/g	61.3	1.2	4.5	2.3
Ti	µg/g	268	15	34	22
Tm [k]	µg/g	4.10	0.08	0.19	0.12
U [l]	µg/g	37.0	1.0	2.2	1.0
W [m]	%	0.338	0.011	0.018	0.010
Yb [n]	µg/g	28.8	0.6	1.5	0.8
Zn	%	0.566	0.009	0.032	0.012
Zr	µg/g	134	4	11	6

***a** sets using digestion by two acids (hydrochloric and nitric), three acids (hydrochloric, nitric and hydrofluoric) or four acids (hydrochloric, nitric, hydrofluoric and perchloric) were excluded as method outliers based on statistical tests*

***b** sets using digestion by two acids were excluded as method outliers based on statistical tests*

***c** methods using digestion by four acids, various fusions and instrumental neutron activation analysis were used by the laboratories*

***d** methods using digestion by four acids and various fusions were used by laboratories*

***e** methods using digestion with four acids and fusion with sodium pyrosulphate were used by the laboratories*

***f** methods using digestion by perchloric acid, four acids in a closed vessel, various fusions and pressed powder followed by X-ray fluorescence were used by the laboratories*

***g** methods using digestion by four acids, various fusions, instrumental neutron activation analysis and pressed powder pellet followed by X-ray fluorescence were used by the laboratories*

***h** methods using digestion by four acids, fusion with lithium metaborate and instrumental neutron activation analysis were used by the laboratories*

***i** methods using various fusions and pressed powder pellet followed by X-ray fluorescence were used by the laboratories* *cont'd*

j methods using four acid digestion in a closed vessel, various fusions and instrumental neutron activation analysis were used by the laboratories
k sets using digestion with four acids in a closed vessel and various fusions were included based on statistical tests
l the only set using digestion with two acids was excluded as a method outlier based on statistical tests and no sets were received using three acids
m almost all sets using digestion with acids were excluded as outliers based on statistical tests
n sets by digestion using four acids were excluded based on statistical tests

Table 2 – MP-2a Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
As	%	0.558	0.010	0.039	0.016
Er [a]	µg/g	22.8	0.4	1.9	1.0
Eu [a]	µg/g	0.1050	0.0070	0.0099	0.0081
Ga [a]	µg/g	26.2	0.6	3.0	1.7
Ho [a]	µg/g	7.04	0.11	0.65	0.35
In [b]	µg/g	12.09	0.20	0.37	0.40
K	%	1.226	0.015	0.070	0.036
Ni [c]	µg/g	9.8	0.8	3.1	1.6
Sb [d]	µg/g	7.84	0.51	0.88	0.47
Te	µg/g	5.75	0.43	0.43	0.39
Tl	µg/g	3.16	0.08	0.76	0.49
Y [e]	µg/g	229	4	16	8

a methods using digestion by four acids (nitric, hydrochloric, hydrofluoric and perchloric) and various fusions were used by laboratories
b statistical analysis of the data warrants classification as provisional despite only 6 sets of data
c data fulfilled the conditions for certified but the element was reclassified as provisional since the between-laboratory standard deviation is approximately 20% of the mean and a considerable amount of the data has only one significant figure
d 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
e sets using digestion by four acids and various fusions were included only based on statistical tests

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

Analyte	Units	Mean	No. accepted laboratories / values
Al AD3a + AD4 [a]	%	3.7	4 / 20
Au [b]	µg/g	0.06	4 / 20
C	%	0.04	7 / 35
Ge	µg/g	8	6 / 30
loss on ignition [c]	%	4	5 / 25
moisture [d]	%	0.1 - 0.5	10 / 45
Na	%	0.03	7 / 35
P	µg/g	90	9 / 45

***a** sets using digestion by three acids (hydrochloric, hydrofluoric and perchloric) or four acids (hydrochloric, nitric, hydrofluoric and perchloric) were included only*
***b** sets using digestion with two acids (hydrochloric and nitric), instrumental neutron activation analysis and fire assay preconcentration were included*
***c** only 5 labs submitted data and only one of which provided full details: ignition of 1 g for 1 hour at 1000°C*
***d** a range is given due to the lack of agreement in the data*

SOURCE
MP-2a is a tungsten-molydenum ore obtained from the Mount Pleasant property in New Brunswick, Canada. The raw material for MP-2a was donated by Adex Minerals Corp and was obtained from the same source as its predecessor, MP-2.

DESCRIPTION
The mineral species include: quartz (64.9%), topaz (6.3%), clinochlore (5.1%), fluorite (5.1%), muscovite (5.1%), almandine (3.3%), orthoclase (3.0%), plagioclase (1.9%), sphalerite (0.8%), wolframite (0.8%), arsenopyrite (0.7%), other (0.6%), epidote (0.5%), Fe-alloy (0.3%), pyrite (0.3%), galena (0.3%), calcite (0.2%), loellingite (0.2%), magnetite (0.2%), molybdenite (0.2%), monazite (0.1%), bismuth (0.07%), cassiterite (0.05%), chalcopyrite (0.04%), euxenite (0.04%), rutile (0.03%), zircon (0.03%) and keiviite (0.02%).

INTENDED USE
MP-2a is suitable for the analysis of tungsten, molybdenum, bismuth, the rare earth elements 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
MP-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 crushed, ground and sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction was 81%. 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 bismuth, copper and molybdenum and inductively coupled plasma – mass spectrometry for silver. 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 bismuth, copper, molybdenum and silver.

CERTIFIED VALUES

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

Methods for the determination of the elements included digestion with perchloric acid, various combinations of acids on a hot plate or 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 and instrumental neutron activation analysis were 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, as well as combustion followed by infrared spectrometry.

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. Forty-three parameters were certified (see Table 1). Many certified elements exclude digestion by two, three and/or four acids as the laboratories did not use this type of preparation, or 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

Twelve 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 indium. Informational values for 8 parameters, shown in Table 3, were derived from the means of a minimum of 3 sets of data.

TRACEABILITY

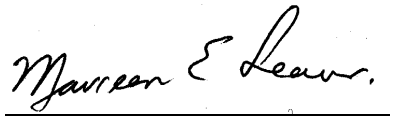
The values quoted herein are based on the consensus values derived from the statistical analysis of the data from the interlaboratory measurement program, and the standards used by the individual laboratories. The report gives the available details.

CERTIFICATION HISTORY
MP-2a was released as a new material in November 2012.

PERIOD OF VALIDITY
The certified values are valid until November 30, 2033.

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



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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.