CCRMP
Canadian Certified Reference Materials Project

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Projet canadien de matériaux de référence certifiés

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Certificate of Analysis

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MP-1b

Certified Reference Material for a Zinc-Tin-Copper-Lead Ore

Table 1 – MP-1b Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	47.0	1.4	3.5	1.8
As	%	2.30	0.04	0.10	0.06
Bi	%	0.0954	0.0023	0.0066	0.0042
Ca	%	2.47	0.05	0.15	0.09
Cd	%	0.0527	0.0015	0.0034	0.0021
Cu	%	3.069	0.042	0.089	0.044
Fe	%	8.19	0.09	0.25	0.14
Mg	%	0.024	0.003	0.006	0.004
Mo*	µg/g	285	10	14	10
Pb	%	2.091	0.029	0.072	0.034
S	%	13.79	0.18	0.40	0.25
Si	%	16.79	0.30	0.60	0.43
Sn	%	1.61	0.045	0.098	0.063
Zn	%	16.67	0.14	0.22	0.12

** two acid digestions removed as method outliers based on statistical tests*

Table 2 – MP-1b Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al_{FUS}*	%	3.465	0.061	0.040	0.043
In	µg/g	565	10	50	37
Sb	µg/g	54.0	1.6	7.9	6.5
Ti_{FUS}*	%	0.0752	0.0036	0.0050	0.0059

** various types of fusions*
For titanium the statistical analysis of the results warrants classification as Provisional, despite only 6 sets

Table 3 – MP-1b Informational Values

Element	Units	Mean	Accepted laboratories / values
Al_{AD}*	%	1.4	9/35
C**	%	0.028	6/24
Co	µg/g	4	3/13
Eu	µg/g	1	3/15
Hf	µg/g	6	3/15
K	%	0.2	3/15
loss on drying	%	0.2	6/22
Lu	µg/g	4	3/15
Mn	µg/g	480	4/20
P	%	0.02	3/15
Sc	µg/g	3	3/15
Ti_{AD}*	%	0.03	4/14
Tb	µg/g	5	3/15
Th	µg/g	50	3/15
U	µg/g	20	3/15
W	µg/g	1100	6/23
Zr	µg/g	150	3/15

** two to four acid digestions*
*** all sets by combustion – infrared spectroscopy*

SOURCE
MP-1b is a zinc-tin-copper-lead ore from Mount Pleasant mine property, New Brunswick, Canada. The raw material was donated by Adex Minerals Corporation, and was obtained from the same mine as its predecessors, MP-1 and MP-1a, which are no longer available.

DESCRIPTION
The mineral species include: quartz (26.0%), sphalerite (23.7%), clinochlore (8.9%), chalcopyrite (7.2%), almandine (6.1%), arsenopyrite (4.3%), fluorite (3.8%), topaz (3.4%), galena (2.4%), cassiterite (2.3%), wolframite (2.2%), pyrite (2.1%), calcite (1.6%), pyrophyllite (1.5%), natrojarosite (1.4%), tennantite

(0.9%), K-feldspar (0.7%), magnetite (0.4%), kaolinite (0.4%), loellingite (0.2%), lepidocrocite (0.2%), and bismuthinite, molybdenite, rutile, scorodite, stannite and zircon, all at 0.1%.

INTENDED USE
MP-1b is suitable for the analysis of zinc, tin, copper, lead and various other elements at major, minor and trace levels in ores. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE
MP-1b should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. The contents of the bottle should be exposed to air for the shortest time possible. Unused material should be stored under an inert gas in a desiccator, or in a new, heat-sealed laminated foil pouch. The values herein pertain to the material when produced. CANMET-MMSL 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 74 µm fraction. The product was blended, and then bottled in 200-gram units. Each bottle was purged with nitrogen and sealed in a laminated polyethylene - foil pouch. The recovery was 75%.

HOMOGENEITY
The homogeneity of the stock was investigated using twenty-two bottles chosen according to a stratified random sampling scheme. Two splits were analyzed from each bottle. The splits were analyzed for bismuth, copper, iron, lead and silver by the digestion of 0.25-gram sample using hydrochloric, nitric, hydrofluoric and perchloric acids followed by inductively coupled plasma – optical emission spectroscopy. Sulphur in 0.15-gram samples was analyzed using a combustion apparatus with infrared detection. Use of a smaller sub-sample than specified above for these elements 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, iron, lead, silver and sulphur.

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

The concentration of the elements was determined by various methods including: multi-acid and microwave digestions, fusions, precipitation, and followed by atomic absorption spectroscopy, flow injection mass spectrometry, hydride generation atomic absorption spectroscopy, inductively coupled plasma – atomic emission spectroscopy, inductively coupled plasma - mass spectrometry, gravimetric analysis and titrations. Fused pellets were used for X-ray fluorescence spectrometry. Combustion - infrared spectroscopy was used for carbon and sulphur. Also, instrumental neutron activation analysis was used for some elements. Cold vapour separation was used for mercury.

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 statistical criterion regarding the agreement of the results. Fourteen elements were certified (see Table 1).

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

document, “Assessment of laboratory proficiency using CCRMP reference materials”, at www.ccrmp.ca under Publications, which is based on Guide 33:2000.

UNCERTIFIED VALUES
Provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CCRMP statistical criterion regarding agreement; or alternatively, 10 or more sets of data, that do not fulfill the CCRMP statistical criteria required for certification. Aluminum by fusion methods, antimony and indium fulfilled these criteria. Additionally, the statistical analysis of the data warranted provisional status for titanium by fusion, despite fewer sets. Informational values for 17 elements, shown in Table 3, were derived from the means of a minimum of 3 sets of data.

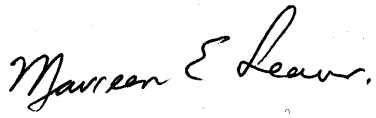
TRACEABILITY
The values quoted herein are based on the consensus values derived from the statistical analysis of the data from the interlaboratory measurement program, and the standards used by the individual laboratories. The report gives the available details.

CERTIFICATION HISTORY
MP-1b is a new material.

PERIOD OF VALIDITY
The certified values are valid until July 31, 2030. The stability of the material will be monitored every two years for the duration of the inventory. Updates will be made via the CCRMP web site.

LEGAL NOTICE
CANMET-MMSL 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 CANMET-MMSL 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 MP-1b 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.