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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CPB-2

Certified Reference Material for Lead Concentrate

Table 1 – CPB-2 Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al	**%**	0.074	0.005	0.013	0.010
Cd	**%**	0.0167	0.0011	0.0017	0.0013
Cu	**%**	0.1213	0.0031	0.0059	0.0040
Fe	**%**	7.065	0.043	0.074	0.056
Mg	**%**	0.0683	0.0035	0.0059	0.0042
Pb	**%**	63.52	0.10	0.10	0.06
Zn	**%**	6.04	0.08	0.13	0.08

Table 2 – CPB-2 Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	**µg/g**	357.3	2.2	4.5	4.0
Bi	**µg/g**	211.2	7.9	9.7	8.3
Ca*	**%**	0.0776	0.0037	0.0052	0.0053
Hg*	**µg/g**	10.03	0.32	0.54	0.52
Mn*	**%**	0.0395	0.0014	0.0037	0.0036
S**	**%**	17.82	0.20	0.52	0.35
S***	**%**	18.22	0.09	0.24	0.26
Sb	**%**	0.423	0.009	0.008	0.008
SiO_2	**%**	0.652	0.023	0.077	0.061

* *Statistical analysis of the results warrants classification as Provisional, despite only 7 sets*
** *Gravimetric and non-gravimetric methods*
*** *Gravimetric method only, statistical analysis of the results warrants classification as Provisional, despite only 6 sets.*

Table 3 – CPB-2 Informational Values

Element	Units	Mean	Number of accepted laboratories / values
As	**%**	0.04	13/56
Au	**µg/g**	0.02	2/6
Ba	**µg/g**	7	2/5
Co	**µg/g**	4	3/10
Cr	**µg/g**	60	5/20
LOD *	**%**	0.3	7/30
K	**%**	0.02	6/21
Mo	**µg/g**	9	3/10
Na	**%**	0.01	6/22
Ni	**µg/g**	11	3/13
Se	**µg/g**	10	3/15
Sn	**%**	0.01	3/15
Tl	**µg/g**	340	6/25

** Loss on drying at 105°C for varying sample masses and times*

SOURCE
CPB-2 is a lead flotation concentrate from the former Sullivan Mine concentrator at Kimberley, British Columbia, Canada. CPB-2 was obtained from the same mine as its predecessor, CPB-1, which is no longer available.

DESCRIPTION

The mineral species include galena (64.7%), anglesite (12.1%), sphalerite (10.1%), pyrrhotite (6.8%), pyrite (4.9%), various silicates (0.6%), quartz (0.3%), biotite (0.2%), chalcopyrite (0.1%) and apatite (0.1%).

INTENDED USE

CPB-2 is suitable for the analysis of lead and various other elements at major, minor and trace levels in lead concentrates. Examples of intended use include quality control, method development and the calibration of equipment.

INSTRUCTIONS FOR USE

CPB-2 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. Changes in the moisture content, caused by the adsorption or loss of moisture, can significantly affect the concentration of lead. 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 dried in an oven at 32°C for 16 hours, crushed, ground, and sieved to remove the plus 74 µm fraction. The product was blended, and then bottled in 200-gram units. The yield was 93%. Each bottle was purged and sealed under nitrogen in a laminated aluminum foil-mylar pouch to prevent oxidation.

HOMOGENEITY

The homogeneity of the stock for lead was investigated using twenty-two bottles chosen according to a stratified random sampling scheme. Two splits were analysed from each bottle. The samples were analyzed according to ISO 13545:2000[1] by a private laboratory that is accredited by the Standards Council of Canada under CAN-P-1579 and CAN-P-4E(ISO/IEC 17025:2005). Samples of approximately 0.3 grams were digested with nitric and sulphuric acids and bromine. Lead was separated from the interfering elements by precipitation as lead sulphate. The precipitate was dissolved in ammonium acetate. Lead was titrated with ethylenediaminetetraacetate using xylenol orange as the indicator.

The homogeneity of the stock for antimony, copper, thallium and zinc was investigated using 16 bottles chosen according to a stratified random sampling scheme. Three splits were analyzed from each bottle. Antimony and thallium were determined in the same 0.1-gram sample using microwave digestion followed by inductively coupled plasma – mass spectrometry. The copper and zinc were determined in the same 0.25-gram sample using a four acid digestion followed by inductively coupled plasma – optical emission spectroscopy. This phase of the work was performed by CANMET-MMSL.

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) was used to assess the homogeneity of these elements[2]. The ratio of the between-bottles to within-bottle mean squares was compared to the F statistic at the 95% level of probability. No evidence of inhomogeneity was observed for antimony, copper, lead, thallium or zinc.

CERTIFIED VALUES

Seventeen industrial, commercial, and government laboratories participated in an interlaboratory measurement program using methods of their own choosing. Lead was determined in some cases according to ISO 13545, and also by a variety of acid digestions both with and without bromine, or fusion; followed by titration, inductively coupled plasma – optical emission spectroscopy, flame atomic absorption spectroscopy or X-ray fluorescence.

ANOVA was used to calculate the consensus values and other statistical parameters[2] 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. Seven elements were certified (see Table 1).

Other elements were determined by various multi-acid digestions, microwave digestion, fusions, drying or combustion; and followed by inductively coupled plasma – optical emission spectrometry, inductively coupled plasma – mass sprectrometry, X-ray fluorescence, gravimetric methods, flame atomic absorption, titrations, or cold vapour analysis.

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

Four 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. Additionally, the statistical analysis of the data warranted provisional status, despite fewer sets, for calcium, manganese, mercury, and sulphur by the gravimetric method only. Informational values for 13 elements, shown in Table 3, were derived from the means of a minimum of 2 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

CPB-2 is a new material.

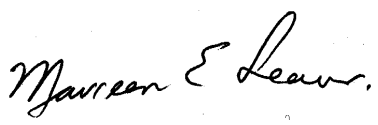
PERIOD OF VALIDITY

The certified values are valid until March 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 CPB-2 Certification Report is available free of charge upon request to:

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REFERENCES

1. ISO 13545:2000 (E), Lead sulfide concentrates – determination of lead content – EDTA titration method after acid digestion, www.iso.org.
2. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960.