Certificate of Analysis

First issued: March 2020 Version: March 2020

CPB-3

Certified Reference Material for a Lead Concentrate

Table 1 – CPB-3 Certified Values

The certified, provisional and indicative values herein pertain to the material on an as-received basis. The exception is lead which has values for both as received basis and dry-mass correction. Values for the elements were generally derived from a variety of digestions and other preparations, followed by instrumental analysis. For some elements, the footnotes indicate further details of the analytical methods used to determine the values. For more detailed information, please refer to the certification report.

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag [a]	µg/g	2790	11	18	12
Al	%	0.203	0.005	0.011	0.006
Ca	%	0.059	0.004	0.010	0.006
Cd	%	0.0652	0.0010	0.0027	0.0013
Co	µg/g	13.6	0.6	1.1	0.7
Cr	%	0.0102	0.0006	0.0016	0.0010
Cu	%	0.240	0.003	0.012	0.005
Fe	%	8.45	0.07	0.21	0.09
Hg [b]	µg/g	40.8	1.2	1.2	0.7
Mg	%	0.1062	0.0025	0.0056	0.0029

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Table 1 – CPB-3 Certified Values *cont'd*

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ni	µg/g	16.8	0.8	3.1	2.2
Pb as received, classical [c]	%	57.94	0.09	0.20	0.10
Pb dry mass - correction, calculated, classical [d]	%	58.02	0.09	0.21	0.10
Pb as received, instrumental [e]	%	58.53	0.33	0.90	0.65
Sb	%	0.580	0.010	0.019	0.010
SiO_2	%	2.62	0.04	0.17	0.09
Zn	%	5.96	0.05	0.14	0.06

a Only data derived from fire assay was included, based on statistical tests.
b All data was derived from various types of acid digestions only.
c Only data derived from ISO 13545:2000, and various similar digestion and titration methods was included, based on statistical tests.
d Only data derived from ISO 13545:2000, and various similar digestion and titration methods was included, based on statistical tests. The data was corrected for moisture.
e Only data using (i) various acid digestions followed by atomic absorption spectrometry or inductively coupled plasma – optical emission spectrometry, or (ii) fusion or fused pellet followed by X-ray fluorescence was included, based on statistical tests.

Table 2 – CPB-3 Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
As	%	0.0391	0.0008	0.0039	0.0020
Au [a]	µg/g	0.119	0.009	0.011	0.012
C [b]	%	1.037	0.029	0.029	0.018

cont'd

Table 2 – CPB-3 Provisional Values *cont'd*

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Mn	%	0.421	0.004	0.021	0.010
S [c]	%	17.03	0.09	0.49	0.23

a The data was derived from various fire assay pre-concentration methods using samples of 10 to 13 grams; statistical analysis of the data warrants classification as provisional despite only 6 sets of data.

b The data was derived mainly from a combustion - infrared spectrometer.

c The data was derived from gravimetric methods, combustion followed by infrared spectrometry and various acid digestions followed by inductively coupled plasma - optical emission spectrometry.

Table 3 – CPB-3 Indicative Values (semi-quantitative only)

Analyte	Units	Mean	No. accepted laboratories / values
Ag [a]	µg/g	2850	6 / 30
Ba	µg/g	60	5 / 22
K	%	0.09	11 / 53
La [b]	µg/g	2	4 / 13
Moisture [c]	%	0.15	16 /72
Na	%	0.01	5 / 22
Sn [b]	µg/g	6	6 / 20
Sr	µg/g	3	5 / 20
Th	µg/g	0.4	4 / 13
Y	µg/g	2	5 / 20

a The data was derived from various types of acid digestion and followed by various instruments, and fusion followed by X-ray fluorescence.

b The data was derived from various types of acid digestions only.

c The data was derived from samples of 1 to 20 grams dried for 1 to 8 hours at 105°C.

SOURCE
CPB-3 is a certified reference material for a lead concentrate donated by a North American refinery.

DESCRIPTION
The mineral species include: anglesite (3.9%), galena (65.5%), hematite (3.9%), pyrite (9.2%), quartz (2.0%), siderite (5.6%), other silicates (0.9%), sphalerite (8.7%), and tetrahedrite – tennantite (0.4%).

INTENDED USE
CPB-3 is suitable for the analysis of lead concentrates for lead and other elements at concentrations ranging from major, minor to trace levels. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE
CPB-3 should be used "as is" without drying for all elements. 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 under an inert gas. Changes in the moisture content, caused by the adsorption or loss of moisture, can significantly affect the concentration of lead.

For applications related to commercial exchange, lead values are generally reported on a dry-mass basis. Separate portions for both the moisture determination and the lead determination should be taken at the same time. ISO 9599:2015, Copper, lead, zinc and nickel sulfide concentrates - Determination of hygroscopic moisture content of the analysis sample - Gravimetric method, or a similar standard, describes the procedure.

The values herein pertain to the date when issued. 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 dried for 24 hours at 32°C, milled and sieved. The recovery of the minus 75 µm (200 mesh) fraction was 81%. The product was blended and bottled in 100-gram units. This is the only size that is available. Each bottle was purged with nitrogen and sealed in a laminated aluminum foil pouch to prevent oxidation.

HOMOGENEITY
The homogeneity of the stock was investigated using fifteen bottles chosen according to a stratified random sampling scheme. Three subsamples were analyzed for lead from each bottle using a method similar to ISO 13545:2000, Lead sulfide concentrates - Determination of lead content - EDTA titration method after acid digestion. After fusion of the acid insoluble residue, atomic absorption spectrophotometry was used to determine the lead. Samples of approximately 0.5 grams were used in each determination. The homogeneity of the stock for cadmium, copper and zinc was investigated using another set of 15 bottles selected according a stratified random sampling scheme. Each sub-sample of 0.5 grams was digested with hydrochloric, nitric, hydrofluoric and perchloric acids followed by inductively coupled plasma – optical

emission spectrometry. A private laboratory that is accredited by the Standards Council of Canada under ISO/IEC 17025:2017 performed these analyses.

A one–way analysis of variance technique (ANOVA)[1] and statistical analyses were used to assess the homogeneity of these elements. No significant between-bottles variation was observed for all elements.

Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters.

CERTIFIED VALUES

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

Carbon was determined using combustion followed by infrared spectrometry.

Chlorine was determined by digestion using various acids or fusion followed by titration, gravimetric analysis, ion chromatography or X-ray fluorescence spectroscopy.

Fluorine was determined by fusion or fused pellet followed by ion selective electrode, ion chromatography or X-ray fluorescence spectroscopy.

Gold was determined by fire assay followed by flame atomic absorption spectroscopy and inductively coupled plasma – optical emission spectroscopy.

Lead was determined by (i) ISO 13545:2000, and various similar methods for digestion, fusion and titration, (ii) digestion using a variety of acids and fusion followed by flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry, and (iii) fused pellet, fusion or pressed powder followed by X-ray fluorescence spectroscopy.

Mercury was determined using digestion using various acids on a hot plate or microwave oven followed by cold vapour atomic absorption spectroscopy, flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy or inductively coupled plasma – mass spectrometry.

Moisture was determined on samples ranging from 1 to 20 grams, dried for 1 to 8 hours at 105ºC.

Silver was determined by fire assay or various acid digestions; followed by gravimetric analysis, flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma - mass spectrometry.

Sulphur was determined using combustion followed by infrared spectrometry; and digestion with various combinations of acids or fusion or fused pellet followed by gravimetric analysis, inductively coupled plasma – atomic emission spectroscopy or X-ray fluorescence spectroscopy.

Methods for the determination of various other elements included (i) digestion with variety of acids on a hot plate or using a microwave oven, or various types of fusions, followed by flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma - mass spectrometry, or (ii) pressed powder, fused pellet or fusion followed by X-ray fluorescence.

ANOVA was used to calculate the consensus values and other statistical parameters from the data 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. Seventeen means 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 33:2017, and the publication, "Assessment of laboratory proficiency using CCRMP reference materials", at www.cccrmp.ca.

UNCERTIFIED VALUES

Five analytes (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. The statistical analysis of the six sets of data for gold using fire assay fulfilled the criteria for provisional status. Indicative values for 10 elements, shown in Table 3, were derived from the means of a minimum of 4 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-3 was released as a new material in March 2020.

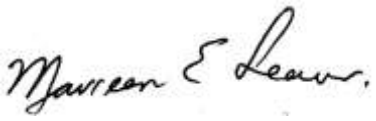
PERIOD OF VALIDITY

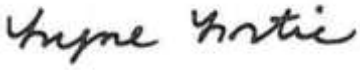
The certified values are valid until March 30, 2040. The stability of the material will be monitored every two years for the duration of the inventory. Updates will be published on the CCRMP web site.

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

CPB-3 was prepared in consideration of the principles in ISO Guides 30, 31, 33 and 35, and ISO 17034. 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.