



CCRMP
Canadian Certified Reference Materials Project



PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

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PTC-1b

Certified Reference Material for Nickel – Copper Sulphide Concentrate with Noble Metals

Table 1 – PTC-1b Certified Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	53.1	1.6	2.2	1.0
As	µg/g	222	8	29	17
Au ^a	µg/g	1.99	0.11	0.18	0.12
Co	%	0.3253	0.0055	0.0082	0.0038
Cu classical ^b	%	7.919	0.025	0.056	0.033
Cu instrumental ^c	%	7.97	0.09	0.17	0.07
Fe	%	36.78	0.45	0.67	0.28
moisture ^d	%	0.81	0.03	0.10	0.07
Mg no AD2,3 ^e	%	0.441	0.008	0.027	0.014
Ni classical ^f	%	11.256	0.036	0.092	0.059
Ni instrumental ^c	%	11.29	0.16	0.21	0.10
Pb	%	0.0795	0.0014	0.0029	0.0015
Pd ^a	µg/g	9.46	0.24	0.25	0.20
Pt ^a	µg/g	6.47	0.24	0.44	0.29
S ^g	%	29.95	0.24	0.64	0.27
Si ^h	%	2.468	0.055	0.065	0.044
Zn	%	0.2083	0.0044	0.0063	0.0038

cont'd

Table 1 – PTC-1b Certified Values *cont'd*

- a most sets involve some type of fire assay pre-concentration*
- b the data includes sets using titration and electrogravimetric methods*
- c the data includes sets using instrumental techniques such as atomic absorption spectrometry, inductively coupled plasma - optical emission spectrometry, inductively coupled plasma – mass spectrometry, X-ray fluorescence, and, for copper only, instrumental neutron activation analysis*
- d samples of 1 to 10 grams were dried for 1 to 60 hours at 100 to 110°C*
- e sets using digestion by two acids (hydrochloric and nitric) or three acids (hydrochloric, nitric and hydrofluoric) were excluded as method outliers based on statistical tests*
- f the data includes sets using titration, electrogravimetric and dimethylglyoxime methods*
- g digestion using four acids (hydrochloric, nitric, hydrofluoric and perchloric), various fusion and combustion methods were used*
- h all sets used a fusion for the preparation*

Table 2 – PTC-1b Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al ^a	%	0.7518	0.0059	0.0059	0.0048
Ba ^{a, b}	µg/g	61.5	2.0	2.0	2.3
Ca ^c	%	0.571	0.017	0.083	0.044
Mn no AD2, 3 ^d	%	0.0193	0.0003	0.0021	0.0018
loss on ignition ^e	%	13.44	0.13	0.49	0.38
Se ^{a, b}	µg/g	120	4	10	11
Ti ^a	%	0.0696	0.0023	0.0047	0.0041

- a digestion using four acids (hydrochloric, nitric, hydrofluoric and perchloric), fusions and instrumental neutron activation analysis were used*
- b statistical analysis of the data warrants classification as provisional despite only 6 sets of data*
- c the only set using digestion by two acids (hydrochloric and nitric) was excluded as a statistical outlier*
- d sets using digestion by two acids or three acids (hydrochloric, nitric and hydrofluoric) were excluded as method outliers based on statistical tests*
- e samples of 0.5 to 2 grams were ignited for 1 to 30 hours at 1000 to 1050°C*

Table 3 – PTC-1b Informational Values (semi-quantitative only)

Element	Units	Mean	No. accepted laboratories / values	Element	Units	Mean	No. accepted laboratories / values
Cd	µg/g	38	7 / 29	Sb ^b	µg/g	6	3 / 11
Cr ^a	%	0.004	6 / 30	Sn ^b	µg/g	120	3 / 13
Ir	µg/g	0.2	3 / 15	Sr ^b	µg/g	30	4 / 14
K ^b	%	0.15	5 / 23	Te ^b	µg/g	30	3 / 11
Mo ^c	µg/g	11	4 / 16	V ^b	µg/g	20	4 / 18
Na ^b	%	0.17	6 / 28	Y ^b	µg/g	3	3 / 11
Rh ^d	µg/g	0.5	3 / 15				

a microwave digestion with two acids (hydrochloric and nitric), digestion with four acids (hydrochloric, nitric, hydrofluoric and perchloric) and instrumental neutron activation analysis were used

b digestion using four acids, fusions and instrumental neutron activation analysis were used

c digestion with three (hydrochloric, nitric and hydrofluoric) or four acids and fusion were used

d fire assay pre-concentration was used for all sets

SOURCE

PTC-1b is a nickel-copper sulphide concentrate with noble metals donated by Vale from its Clarabelle mill in Copper Cliff, Ontario, Canada. This is the same source as its predecessor, PTC-1a.

DESCRIPTION

The mineral species include: pentlandite (34.7%), chalcopyrite (23.2%), pyrrhotite (22.4%), pyrite (5.6%), anorthoclase (4.9%), Fe-Si-S-O (possibly danalite, 3.6%), Fe-Ni alloy (1.0%), bravoite (0.8%), magnetite (0.6%), biotite and clinocllore (both at 0.5%), gypsum, millerite and quartz (all at 0.4%), other and sphalerite (both at 0.2%), ilmenite (0.1%).

INTENDED USE

PTC-1b is suitable for the analysis of nickel, copper, gold, platinum and palladium and other elements in concentrates in concentrations ranging from major, minor to trace levels. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE

PTC-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. 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 at 32°C and sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction from the dried material was 61%. 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 to prevent oxidation. 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. Nickel was determined in subsamples of 1 gram by acid digestion, separation using dimethylglyoxime, and electroplating with correction for co-plating elements. Copper was determined in subsamples of 1 gram by acid digestion and electroplating with correction for co-plating elements. Gold, platinum and palladium were determined in subsamples of 5 grams using fire assay pre-concentration followed by determination using inductively coupled plasma – optical emission spectrometry.

An additional three subsamples were taken from each of the 15 randomly chosen bottles. Subsamples of 0.25 grams were digested using hydrochloric, nitric, hydrofluoric and perchloric acids, and analyzed by inductively coupled plasma – optical emission spectrometry for cobalt, and inductively coupled plasma – mass spectrometry for lead. Samples of 0.1 grams were digested with hydrochloric and nitric acids in a microwave and analyzed by inductively coupled plasma – mass spectrometry for arsenic. Samples of 0.1 grams were analyzed for sulphur using a combustion apparatus with infrared detection. 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 or statistical analyses were used to assess the homogeneity of these elements. No significant between-bottle variation was observed for all elements.

CERTIFIED VALUES

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

Classical methods for nickel included titration, dimethylglyoxime and electrogravimetric methods. Classical methods for copper included titration with thiosulphate, other titration methods and electrogravimetric methods.

Gold, platinum and palladium were determined by various fire assay pre-concentration methods including both nickel sulphide collection and lead button collection followed by determination using inductively coupled plasma – optical emission spectrometry and inductively coupled plasma – mass spectrometry.

Methods for the determination of the elements included digestion with various combinations of acids with and without the addition of bromine 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 fused pellet and instrumental neutron activation were also used for many elements.

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

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. Seventeen analytes 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 publication, "Assessment of laboratory proficiency using CCRMP reference materials", at www.ccrmp.ca.

UNCERTIFIED VALUES

Seven 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 sets of data for which the statistical analysis of the data warranted provisional status. This latter group includes barium and selenium. Informational values for 13 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

PTC-1b was released as a new material in May 2014.

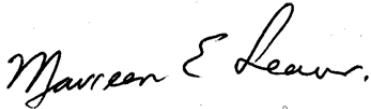
PERIOD OF VALIDITY

The certified values are valid until May 31, 2034. 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

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.