



CCRMP
Canadian Certified Reference Materials Project

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Certificate of Analysis

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SU-1b

Certified Reference Material for a Nickel-Copper-Cobalt Ore

Table 1 – SU-1b Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	6.39	0.34	0.21	0.12
Al*	%	4.30	0.07	0.20	0.10
As	µg/g	2.49	0.33	0.58	0.40
Ca*	%	2.21	0.03	0.11	0.06
Co	%	0.0672	0.0013	0.0034	0.0013
Cu	%	1.185	0.019	0.037	0.014
Fe	%	25.54	0.30	0.68	0.27
Mg*	%	1.790	0.026	0.098	0.051
Mn*	%	0.0703	0.0010	0.0028	0.0019
Ni	%	1.953	0.029	0.044	0.017
Pb	µg/g	58	3	10	5
Pd	µg/g	0.791	0.038	0.040	0.025
Pt	µg/g	0.491	0.042	0.035	0.022
S	%	14.14	0.21	0.23	0.11
Si**	%	15.23	0.25	0.31	0.22
Zn	µg/g	235	9	30	19

* excludes digestions with two acids (usually nitric and hydrochloric) as statistical outliers

** fusions only



Table 2 – SU-1b Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Bi*	µg/g	2.73	0.50	0.63	0.70
Se	µg/g	20.7	0.7	4.3	2.3
V*	µg/g	82.5	2.3	4.7	4.4

* *statistical analysis of the results warrants classification as provisional, despite only 6 data sets for bismuth and 7 data sets for vanadium.*

Table 3 – SU-1b Informational Values

Element	Units	Mean	No. accepted laboratories / values	Element	Units	Mean	No. accepted laboratories / values
Au	µg/g	0.2	17 / 80	LOI*	%	8	3 / 15
Ba	µg/g	350	3 / 14	Lu	µg/g	0.09	3 / 15
Be	µg/g	0.4	3 / 15	Mo	µg/g	4	5 / 25
C	%	0.04	3 / 14	Na	%	1.6	5 / 24
Cd	µg/g	3	4 / 20	Nb	µg/g	3	3 / 15
Ce	µg/g	35	4 / 20	Nd	µg/g	15	3 / 15
Cr	µg/g	320	7 / 35	P	%	0.06	5 / 25
Cs	µg/g	0.3	4 / 20	Rb	µg/g	13	4 / 20
Dy	µg/g	1.4	3 / 15	Sb	µg/g	0.2	3 / 15
Er	µg/g	0.7	3 / 15	Sc	µg/g	9	4 / 20
Eu	µg/g	0.7	3 / 15	Sm	µg/g	3	4 / 20
Ga	µg/g	10	4 / 20	Sr	µg/g	280	6 / 29
Gd	µg/g	2	3 / 15	Tl	µg/g	0.3	4 / 20
H ₂ O(105°C)	%	0.6	2 / 10	U	µg/g	0.2	4 / 19
Ho	µg/g	0.3	3 / 15	Y	µg/g	7	4 / 20
K	%	0.6	4 / 18	Yb	µg/g	0.6	4 / 20
La	µg/g	17	4 / 20				

* *loss on ignition*

SOURCE

SU-1b is a nickel-copper-cobalt ore from Copper Cliff, Ontario, Canada. The raw material was donated by Vale Inco Limited, and was obtained from the same mine as its predecessors, SU-1 and SU-1a, which are no longer available.

DESCRIPTION

The mineral species include: pyrrhotite (24.4 %); plagioclase (21.8 %); quartz (11.0 %); magnetite (7.3 %); ferrohornblende (6.7 %); pentlandite (5.4 %); clinocllore (5.2 %); K-feldspar (4.5 %); chalcopyrite (3.3 %); pyrite (3.2 %); enstatite (1.5 %); epidote (1.3 %); augite (1.0 %); biotite (1.0 %); talc (0.6 %); various other minor silicates (0.5 %); actinolite (0.4 %); hypersthene, ilmenite and titanite, all at 0.2 %; and barite, berthierine, dolomite and lizardite, all at 0.1 %.

INTENDED USE

SU-1b is suitable for the analysis of nickel, copper, cobalt, platinum, palladium 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

SU-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 recovery was 82%. 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.

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 cobalt, copper, and nickel by the digestion of 0.25-gram sample using hydrochloric, nitric, hydrofluoric and perchloric acids followed by inductively coupled plasma – optical emission spectroscopy. Gold, platinum and palladium in 15-gram samples were determined using lead fire assay and inductively coupled plasma – optical emission spectroscopy and mass spectrometry. 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 cobalt, copper, nickel, palladium, platinum and sulphur. Gold is suspected of having a nugget effect, and thus was not certified.

CERTIFIED VALUES

Twenty-seven 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 digestions or fusions; followed by atomic absorption spectroscopy, inductively coupled plasma – atomic emission spectroscopy, inductively coupled plasma - mass spectrometry, graphite furnace - atomic absorption spectroscopy, coulometry or gravimetric analysis. Gold, platinum and palladium were concentrated by lead and nickel sulphide fire assay. Fused pellets were used for X-ray fluorescence spectrometry and other finishes. Combustion - infrared spectroscopy was used for sulphur. Also, instrumental neutron activation analysis was used for some elements.

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. Sixteen (16) 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 publication, "Assessment of laboratory proficiency using CCRMP reference materials", at www.ccrmp.ca.

UNCERTIFIED VALUES

The provisional value for selenium (Table 2) was derived from 16 sets of data that did not fulfill the CCRMP statistical criteria required for certification. Additionally, the statistical analysis of the data warranted provisional status for the 6 sets of data for bismuth and 7 sets of data for vanadium. Informational values for 33 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

SU-1b is a new material.

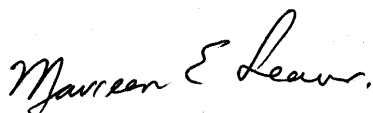
PERIOD OF VALIDITY

The certified values are valid until January 31, 2031. 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

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



Maureen E. Leaver – CCRMP Coordinator



Joseph Salley – Data Processor

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.