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CERTIFICATE OF ANALYSIS

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ACCREDITED
CERTIFICATE #4015.01

CBPA-1

Copper Sulfide Ore (Sossego, Pará)

Original certificate: December, 2007
Revision: September, 2016

The CBPA-1 is a copper sulfide ore sample originating from Sossego region, located in Pará State, Brazil. The raw material was air dried, crushed and pulverized to pass a 0.075 mm screen and then homogenized. This reference material is intended for use in the development, validation and quality control of analytical methods and calibration of measurement equipment for the determination of copper and other constituents in sulfide minerals. A unit of CBPA-1 consists of 135 g of powdered ore packaged under nitrogen atmosphere in a glass bottle and vacuum sealed in a laminated aluminum foil cromopel pouch.

This material was certified by means of an interlaboratory program involving twenty expert laboratories in this field, using methods of their choice. A one-way analysis of variance technique [1] were performed on the data to estimate the overall mean and variability components. The assignment of certified property values was determined by the quality of the data based on such aspects as a minimum of ten accepted sets of data, ratio of the between- to the within variability component less than or equal to 3 and percentage of sets that are rejected do not exceed 15% [2]. The stated uncertainty is an expanded uncertainty, with coverage factor 2, estimated by combining the uncertainty components due to batch inhomogeneity and batch characterization [3].

Certified Values

Constituent	Unit	Mass fraction	Repeatability standard deviation [1]	Between-laboratory standard deviation [1]	No. sets of data	Minimum sample (g)[*1]	Analytical methods
Al_2O_3	% m/m	10.10 ± 0.13	1.8E-01	2.3E-01	14	0.1	a; e; p; s; u; v
CaO	% m/m	2.966 ± 0.068	6.1E-02	1.3E-01	16	0.1	a; e; n; p; t; u
Co	mg/kg	78 ± 2.6	2.8E+00	4.9E+00	15	0.1	a; e; q; u; v
Cr	mg/kg	26 ± 2.6	2.9E+00	3.8E+00	10	0.1	e; p; u; v
Cu	% m/m	0.978 ± 0.013	1.4E-02	3.0E-02	23	0.1	a; e; f; n; p; u; v
Fe_2O_3	% m/m	16.45 ± 0.15	1.9E-01	2.9E-01	17	0.1	a; e; g; n; p; t; u; v
K_2O	% m/m	1.853 ± 0.050	5.7E-02	8.6E-02	13	0.2	b; c; e; p; u; v
MgO	% m/m	3.288 ± 0.034	6.0E-02	5.5E-02	13	0.1	a; e; n; p; u
Mn	% m/m	0.0578 ± 0.0016	1.3E-03	3.2E-03	16	0.1	a; e; n; p; u; v
Na_2O	% m/m	1.423 ± 0.028	3.8E-02	4.9E-02	13	0.2	b; c; e; p; u; v
Ni	mg/kg	276 ± 10	6.8E+00	2.0E+01	15	0.1	a; e; f; p; q; u; v
P_2O_5	% m/m	1.000 ± 0.032	2.7E-02	5.5E-02	12	0.1	d; e; p; s; u
Zn	mg/kg	126 ± 5.4	4.3E+00	1.1E+01	17	0.1	a; e; f; n; q; u; v

[*1] smallest mass sample used in the interlaboratory measurement program.

ADDITIONAL INFORMATION ON COMPOSITION

Noncertified property values are provided for information only. Indicative values were assigned to property values derived from at least eight sets of data that did not fulfill a specific statistical criteria required for certification, but which uncertainty is fit-for-purpose. Informative values were estimated by the median of at least three sets of data.

Indicative Values

Constituent	Unit	Mass fraction	Repeatability standard deviation [1]	Between-laboratory standard deviation [1]	No. of sets of data	Minimum sample (g)[*1]	Analytical methods
Mo	mg/kg	10 ± 3	1.5E+00	4.3E+00	10	0.1	a; e; q; u
Pb	mg/kg	33 ± 5	3.0E+00	6.3E+00	8	0.2	a; e; f; p; q; u
SiO_2	% m/m	56.4 ± 0.5	2.4E-01	8.0E-01	10	0.1	n; o; p; u
Sr	mg/kg	67 ± 2	2.4E+00	5.8E+00	8	0.2	e; q; u
Ti	% m/m	0.35 ± 0.01	5.9E-03	1.9E-02	10	0.1	a; e; n; p; u

[*1]smallest mass sample used in the interlaboratory measurement program.

Informative Values

Constituent	Unit	Mass fraction	Range	No. of data	Analytical methods
As	mg/kg	9	0.8 – 43	30	e; f; u
Au	mg/kg	0.17	0. 09 – 0 5	27	f; k; l; m
Ba	mg/kg	478	457 - 509	15	q; u
C	% m/m	0.16	0.14 – 0.18	30	j; p
Cd	mg/kg	3.0	0.3 - 4	15	a; e; f
Ce	mg/kg	427	399 - 467	35	e; q; u; v
Cu soluble	% m/m	0.5	0.4 – 0.8	29	a; e
F	mg/kg	708	400 - 1103	25	r; s
Ga	mg/kg	22.2	19 - 26	15	q; u
La	mg/kg	267	239 - 322	40	e; p; q; u; v
Nb	mg/kg	10	6 - 17	20	e; q; u
Nd	mg/kg	143	111 - 156	20	q; u; v
Rb	mg/kg	101	81 - 120	35	e; q; u; v
S	% m/m	0.16	0.14 – 0.20	39	i; j; p; u
Sc	mg/kg	16	14 - 22	20	p; u; v
Sm	mg/kg	20	18 - 43	23	q; u; v
Sn	mg/kg	7.0	4 - 11	15	q; u
Ta	mg/kg	5	0.7 - 12	19	e; q; u; v
Th	mg/kg	25	22 - 70	35	e; q; u; v
U	mg/kg	41	33 - 52	35	e; q; u; v
V	mg/kg	168	155 - 188	25	e; q; u
Y	mg/kg	69	48 - 78	20	e; q; u
Zr	mg/kg	149	2 - 167	27	e; p; q; u
Loss of mass, 1000 °C	% m/m	3.8	3.4 – 4.9	45	h

The mineral composition of CBPA-1 was identified by X-ray diffraction (XRD). The major minerals are quartz, amphiboles, chlorite, plagioclase and magnetite. Chalcopyrite, pyrite and fluorapatite were identified as minor minerals.

INSTRUCTIONS FOR USE

Analyses must be performed on samples "as is", without drying. The contents of the bottle should be mixed (by rolling the bottle) before taking samples. The mass of samples used for analyses should be greater than the minimum size indicated for certified and indicative property values. Avoid prolonged exposure to air. Tightly recap the bottle after sampling. Where frequently using, a part of the material must be packaged in a weighing bottle, with a semi-opened lid, and kept in a desiccator under vacuum.

STORAGE

The bottle should be stored in a desiccator under vacuum or vacuum sealed in a laminated aluminum foil pouch to minimize oxidation and moisture absorption.

HAZARDOUS SITUATION

This material contains fine mineral particulate. Avoid dispersion of, exposure to dust by inhalation, eye contact or skin contact. Dispose residual material in accordance with regulations pertaining for inorganic chemical and mineralogical waste.

LEVEL OF HOMOGENEITY

To assess homogeneity, twenty units were selected from the batch of CBPA-1 using a stratified random sampling scheme. For each selected unit, measurements were carried out in triplicate, under repeatability conditions, by pressed pellet (0.5 g of sample) / X-ray fluorescence spectrometry. A one-way analysis of variance approach was performed on the data to compute the within and the between-unit standard deviations. The uncertainty component due to batch inhomogeneity, expressed as a percentage of the certified value, is less than 5 %.

LEVEL OF STABILITY

CBPA-1 is considered to be stable. Based on the nature of the material and previous chemical and mineralogical analysis, deterioration is not anticipated provided the material is handled and stored in accordance with instructions given in this certificate.

METROLOGICAL TRACEABILITY

In the characterization process by an interlaboratory program, the selection of measurement methods as well as respective calibrants was based on the decision of each participating laboratory. A consequence of such an approach is that the metrological traceability chain(s) for each of the assigned values (combined from a number of results) cannot easily be described, but are expected to include independent sources of bias. Therefore the demonstrated agreement of independent measurements resulting from the various methods, calibrants, and validation steps using previously certified materials results in certified values that are metrologically traceable to the SI units of mass and amount of substance.

ANALYTICAL METHODS

a acid digestion / flame atomic absorption spectrometry
b acid digestion / flame emission spectrometry
c acid digestion / flame photometry
d acid digestion / gravimetry
e acid digestion / inductively coupled plasma optical emission spectrometry
f acid digestion / inductively coupled plasma mass spectrometry
g acid digestion / titrimetry
h calcination / gravimetry
i combustion / gravimetry
j combustion / infrared spectrometry
k fire assay / flame atomic absorption spectrometry
l fire assay / gravimetry
m fire assay / inductively coupled plasma optical emission spectrometry
n fusion / flame atomic absorption spectrometry
o fusion / gravimetry
p fusion / inductively coupled plasma optical emission spectrometry
q fusion / inductively coupled plasma mass spectrometry

r fusion / ion selective eletrode
s fusion / spectrophotometry
t fusion / titrimetry
u fused pellet / X-ray fluorescence spectrometry
v instrumental neutron activation analysis

PARTICIPATING LABORATORIES

- Acme Analytical Laboratories Ltd., Vancouver, Canada
- Alfred H. Knight Ltd. International - Minera Valle Central and Faena Los Pelambres Laboratories, Santiago, Chile
- Alfred H. Knight Ltd. International, St. Helens, UK
- ALS Chemex, North Vancouver, Canada
- Anglo Research - Crown Mines, Johannesburg, South Africa
- Bundesanstalt für Geowissenschaften und Rohstoffe - Geochemie, Hannover, Germany
- Canada Center for Mineral and Energy Technology - Mining and Mineral Sciences Laboratories - Analytical Service Group, Ottawa, Canada
- Caraíba Metais S/A - Divisão de Desenvolvimento da Qualidade, Dias d'Ávila, Brasil
- Central Geological Laboratory, Ulaanbaatar, Mongolia
- Centro de Estudios, Medicion y Certificacion de Calidad Ltda - Departamento de Química y Minerales, Santiago, Chile
- Centro de Investigaciones para la Industria Minero-Metalúrgica - Caracterización de Materiales, Ciudad de la Habana, Cuba
- Centro de Tecnologia Mineral - Coordenação de Análises Minerais, Rio de Janeiro, Brasil
- Comisión Chilena de Energía Nuclear - Laboratorio de Análisis por Activación Neutrónica, Santiago, Chile
- Compañía Contractual Minera Candelaria - Departamento Químico, Copiapó, Chile
- Vale – Departamento de Desenvolvimento de Projetos Minerais - Laboratório, Santa Luzia, Brasil
- Vale – Mina do Sossego - Laboratório, Canaã dos Carajás, Brasil
- Eurotest Control JSC, Sofia, Bulgaria
- Set Point Laboratories, Johannesburg, South Africa
- SGS del Peru, Lima, Peru

PERIOD OF VALIDITY

The certified values are valid until December 2029, provided the CBPA-1 unit is handled and stored in accordance with instructions given in this certificate. This certification is nullified if the material is damaged, contaminated or otherwise modified. The stability of CBPA-1 will be monitored over the period of validity. Updates will be published on the CETEM website.

FURTHER INFORMATION

The certification report is available upon request to CETEM. For details on the interpretation of measurement results on CETEM's certified reference materials, access the publication "Application Guide 1" at www.cetem.gov.br/mrc.

CERTIFYING OFFICER

The technical and management aspects involved in the preparation, certification and issuance of the CBPA-1 were coordinated through the CETEM's Certified Reference Material Program.

Maria Alice Goes
Certified Reference Material Program Coordinator

REFERENCES

[1] ISO 5725-5:1998. Accuracy (trueness and precision) measurement methods and results – Part 5: Alternative methods for determination of the precision of a standard measurement method. International Organization for Standardization (ISO), Geneva.
[2] Steger, H.F. "A re-assessment of the criteria for certification in CCRMP"; Geostandards Newsletter 6:1: 17-23; 1982.
[3] ISO Guide 35:2006. Reference materials – General and statistical principles for certification. International Organization for Standardization (ISO), Geneva.