Certificate of Analysis

First issued: May 2011 Version: May 2011

CCU-1d

Certified Reference Material for a Copper Concentrate

Table 1 – CCU-1d Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	120.7	2.2	3.8	1.3
Al no AD2[a]	%	0.191	0.007	0.023	0.012
Au	µg/g	14.01	0.34	0.34	0.18
Ca no AD2[a]	%	0.195	0.008	0.021	0.010
Cd	µg/g	245.9	4.8	17.3	6.3
Co	µg/g	330	8	28	12
Cu classical[b]	%	23.985	0.039	0.098	0.039
Cu instrum[c]	%	23.93	0.29	0.60	0.28
Fe all[d]	%	29.26	0.42	1.21	0.41
Mg no AD2[a]	%	0.508	0.009	0.025	0.011
Mn no AD2[a]	µg/g	99.4	2.7	7.9	4.4
Pb	%	0.262	0.008	0.015	0.005
S all[e]	%	32.76	0.34	0.63	0.25
Sb	µg/g	61.9	3.9	9.7	6.2
SiO_2	%	3.036	0.076	0.098	0.054
Zn	%	2.63	0.05	0.12	0.04

***a** sets using digestion by two acids, hydrochloric and nitric acids, were excluded as method outliers based on statistical tests*

***b** sets using iodometric methods, ISO 10258:1994, titration methods, electrogravimetric methods, ISO 10469:2006 and electrolysis only included*

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c sets using instrumental techniques such as atomic absorption spectrometry, inductively coupled plasma - optical emission spectrometry, inductively coupled plasma - mass spectrometry, and X-ray fluorescence, and instrumental neutron activation analysis only included

d sets by classical and instrumental methods included; Tables 2 and 3 contains means by various methods

e sets by gravimetric and instrumental methods included; Table 2 contains means by each method

Table 2 – CCU-1d Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
As	%	0.0545	0.0011	0.0048	0.0019
Ba[a]	µg/g	12.41	0.82	0.82	0.72
C	%	0.088	0.008	0.024	0.018
Fe FAA[a, b]	%	29.95	0.51	0.51	0.49
Fe ICPE[c]	%	28.1	0.5	2.1	1.2
Fe TITN[d]	%	29.54	0.10	0.43	0.32
Hg [e]	µg/g	7.0	0.7	1.5	0.9
K no AD2[f]	%	0.0316	0.0016	0.0050	0.0042
Loss on ignition[g]	%	19.51	0.11	0.85	0.74
Mo no AD2[f]	µg/g	18.1	0.7	3.0	1.8
Moisture[h]	%	2.03	0.07	0.14	0.12
Ni [i]	µg/g	7.6	0.9	1.6	0.9
S GRV [a, j]	%	32.88	0.07	0.23	0.22
S instrum[k]	%	32.73	0.41	0.75	0.38
Se	µg/g	244	7	37	19
Sr	µg/g	4.9	0.3	1.0	0.8
Te[i]	µg/g	36.7	2.3	7.4	4.3
Ti no AD2[f]	µg/g	66.0	2.3	7.5	5.8
Tl[a]	µg/g	2.63	0.09	0.22	0.23

a statistical analysis of the data warrants classification as provisional despite only 6 or 7 sets of data

b sets by flame atomic absorption spectrometry were included only, based on statistical tests

c sets by inductively coupled plasma - emission spectrometry were included only, based on statistical tests; although the data met the criteria for certification a provisional value was given in order to avoid the impression that inductively coupled plasma - emission spectrometry is more accurate and precise than titration

d sets by titration included only, based on statistical tests

e data fulfilled the conditions for certification, but the element was re-classified as provisional since many sets have only one significant figure

f sets using digestion by hydrochloric and nitric acids were excluded as method outliers based on statistical tests

g samples of 1 to 10 grams ignited for 1 to 30 hours at 400 to 1050°C

h samples of 1 to 10 grams dried for 0.1 to 60 hours at 105 to 110°C

cont'd

i data fulfilled the conditions for certified but the element was reclassified as provisional since the between-laboratory standard deviation is approximately 20% of the mean
j sets using gravimetric analysis only included, based on statistical tests
k sets by inductively coupled plasma - optical emission spectrometry, combustion - infrared spectrometry, combustion - automatic titration, and fusion followed by X-ray fluorescence were included only, based on statistical tests; although the data met the criteria for certification, a provisional value was given in order to avoid the impression that instrumental methods are more accurate and precise than gravimetric analysis

Table 3 – CCU-1d Informational Values (semi-quantitative only)

Element	Units	Mean	No. accepted laboratories / values	Element	Units	Mean	No. accepted laboratories / values
Al AD2[a]	%	0.09	5 / 25	Mg AD2[a]	%	0.11	5 / 25
Bi[b]	µg/g	3	8 / 40	Mn AD2[a]	%	0.008	5 / 25
Ca AD2[a]	%	0.17	5 / 25	Na	%	0.02	11 / 55
Ce AD2[a, c]	µg/g	2	3 / 15	P	µg/g	30	4 / 18
Cl	µg/g	60	3 / 15	Re	µg/g	0.006	3 / 15
Cr	µg/g	6	7 / 35	Sm	µg/g	0.6	3 / 15
Cs AD [d]	µg/g	0.08	3 / 15	Sn	µg/g	12	6 / 30
F	µg/g	200	8 / 39	U	µg/g	0.7	5 / 25
Fe XRF[e]	%	30.	4 / 16	W	µg/g	0.4	3 / 15
In	µg/g	7	4 / 20	Zr no AD2[f]	µg/g	14	5 / 25
La no AD2 [c, f]	µg/g	3	3 / 15				

a sets by digestion using hydrochloric and nitric acids only included
b sets by inductively coupled plasma - mass spectroscopy only included
c data suggests that digestions by two acids may have a lower recovery than other methods
d sets by acid digestions only included
e sets by X-ray fluorescence only included; mean is 30 ± 1
f sets using digestion by hydrochloric and nitric acids were excluded as method outliers

SOURCE
CCU-1d is a copper concentrate from the Flin Flon mill, Manitoba, and donated by Hudson Bay Mining and Smelting Company Limited, Flin Flon, Manitoba, Canada. The raw material for CCU-1d was obtained from the same company as its predecessor, CCU-1c.

DESCRIPTION
The mineral species include: albite (0.1%), anorthite (0.01%), bornite (0.1%), chalcopyrite (66.8%), chalcocite (0.1%), chlorite (0.3%), covellite (0.1%), digenite (0.1%), diopside (0.1%), gypsum (0.3%), magnetite (0.4%), muscovite (0.1%), nontronite (0.3%), pyrite (20.5%), pyrrhotite (3.8%), pyroxene (1.6%), quartz (1.6%) and sphalerite (3.8%).

INTENDED USE
CCU-1d is suitable for the analysis of copper and various elements at major, minor and trace levels in copper concentrates. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE
CCU-1d 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 dried at 33°C, crushed, sieved, mixed and put into bottles each containing 200 grams. The recovery was 67% with a particle size of less than 75 µm (200 mesh). Each bottle was purged with nitrogen and sealed in a laminated polyethylene - 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 from each bottle. Copper in samples of 0.5 grams was analyzed using the short iodide method which includes titration using sodium thiosulphate, and fusion of the acid insoluble material followed by determination using atomic absorption spectroscopy. The gold in samples of 5 grams was concentrated using lead fire assay and determined using a gravimetric analysis.

Each bottle in a second set of 15 randomly chosen bottles was divided into 8 subsamples and three subsamples were analyzed. The sulphur was determined in samples of 150 milligram using combustion followed by measurement by infrared spectroscopy. The zinc in samples of 0.25 grams was determined using a digestion with four acids followed by inductively coupled plasma – optical emission spectrometry. Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters. The evidence indicates that CCU-1d is sufficiently homogeneous for use as a certified reference material.

CERTIFIED VALUES
Forty-one industrial, commercial and government laboratories participated in an interlaboratory measurement program using methods of their own choosing.

Methods for the analysis of copper included various acid digestions or microwave digestions and separations followed by classical measurement methods such as iodometric and various titration methods, ISO 10258 1994, electrogravimetric analysis, electrolysis, gravimetric methods, ISO 10469:2006; and also instrumental techniques such as such as flame atomic absorption spectrometry, inductively coupled plasma – optical emission spectrometry and inductively coupled plasma – mass spectrometry. Fusion followed by X-ray fluorescence was also used.

Fire assay by lead collection followed by gravimetric finish was the most common method for the analysis of gold. Various types of fire assay pre-concentration and a microwave digestion followed by atomic absorption spectrometry, gravimetric analysis, inductively coupled plasma – optical emission spectrometry, inductively coupled plasma – mass spectrometry, and instrumental neutron activation analysis were also used.

The methods used for various other elements included multi-acid digestions with and without the addition of hydrobromic acid, microwave digestion, various types of fusions, and followed by determination using flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry. Various types of fusions or fused pellet were followed by X-ray fluorescence. Instrumental neutron activation analysis was also used. Several elements were analyzed by other methods by some laboratories. Sulphur and silicon were determined by gravimetric analysis. Carbon and sulphur were determined by combustion infrared spectroscopy and combustion-

titration. Cold vapour atomic absorption spectrometry and cold vapour fluorescence spectrometry were used for mercury. Turbidimetric analysis, distillation, visible and ultraviolet spectrometry, ion specific electrode and ion chromatography were used for fluorine and chlorine.

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 means were certified (see Table 1). Many certified elements exclude digestion by two acids based on statistical tests.

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

Nineteen provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CRRMP statistical criterion regarding agreement; or 10 or more sets of data, that do not fulfill the CCRMP statistical criteria required for certification; or 6 or 7 sets of data for which the statistical analysis of the data warranted provisional status. This latter group includes barium, iron by flame atomic absorption spectroscopy, sulphur by gravimetric analysis and tellurium. Informational values for 21 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

CCU-1d is a new material.

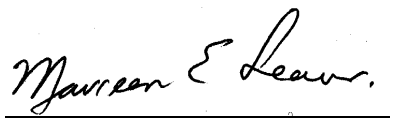
PERIOD OF VALIDITY

The certified values are valid until June 30, 2033. 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



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FOR FURTHER INFORMATION

The Certification Report is available free of charge upon request to:

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