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**CCRMP**

Canadian Certified Reference Materials Project



**PCMRC**

Projet canadien de matériaux de référence certifiés

# Certificate of Analysis

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## CCU-1e

### Certified Reference Material for a Copper Concentrate

**Table 1 – CCU-1e Certified Values**

*note: The values were generally derived from digestion methods using various acids and various complete digestion techniques. The footnotes indicate further details of analytical methods used to determine certified, provisional or indicative values. For more detailed information, please refer to the certification report.*

Element	Units	Mean	Within-lab Standard Deviation	Between- labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	205.2	3.4	4.9	2.0
Al non AD2 <sup>a</sup>	%	0.1385	0.0042	0.0074	0.0033
As	%	0.101	0.003	0.014	0.005
Au <sup>b</sup>	µg/g	20.27	0.52	0.52	0.21
C <sup>c</sup>	%	0.100	0.012	0.012	0.008
Ca	%	0.129	0.004	0.012	0.005
Cd	µg/g	74.2	1.9	6.6	2.6
Co non AD2 <sup>a</sup>	µg/g	301	6	14	6
Cu classical <sup>d</sup>	%	23.07	0.04	0.11	0.05
Cu instrum <sup>e</sup>	%	22.88	0.24	0.51	0.24
Fe all <sup>f</sup>	%	30.7	0.3	1.4	0.5
Hg	µg/g	10.4	0.5	1.6	0.8
Mg	%	0.706	0.009	0.030	0.013
Mn non AD2 <sup>a</sup>	µg/g	96	4	13	7
Pb	%	0.703	0.009	0.031	0.012

*Cont'd*

**Table 1 – CCU-1e Certified Values** *cont'd*

Element	Units	Mean	Within-lab Standard Deviation	Between- labs Standard Deviation	95% Confidence Interval of Mean
S all <sup>g</sup>	%	35.28	0.26	0.47	0.20
S COMB <sup>h</sup>	%	35.52	0.30	0.31	0.22
Sb	µg/g	104	3	16	7
SiO <sub>2</sub> <sup>i</sup>	%	3.134	0.055	0.074	0.039
Te	µg/g	61.8	2.6	9.3	4.7
Tl	µg/g	2.69	0.09	0.16	0.12
Zn	%	3.02	0.03	0.10	0.04

*a data using digestion by two acids, hydrochloric and nitric acids, was excluded as method outlier based on statistical tests*

*b various pre-concentration methods using fire assay and sample weights of 7 to 30 grams were used*

*c mainly combustion apparatus with infrared spectroscopy was used*

*d data using iodometric methods, ISO 10258:1994, titration methods, electrogravimetric methods and gravimetric methods was included only, based on statistical tests*

*e data using instrumental techniques such as atomic absorption spectrometry, inductively coupled plasma – optical emission spectrometry and X-ray fluorescence only was included, based on statistical tests*

*f data by classical and instrumental methods was included; Tables 2 and 3 contain means by various methods*

*g data by a variety of preparation and determination steps was included*

*h data from combustion apparatus only was included, based on statistical tests*

*i mainly fusion methods were used*



**Table 2 – CCU-1e Provisional Values**

Analyte	Units	Mean	Within-lab Standard Deviation	Between- labs Standard Deviation	95% Confidence Interval of Mean
F <sup>a</sup>	µg/g	255	12	24	21
Fe FAA <sup>b, c</sup>	%	31.49	0.30	0.70	0.74
K	%	0.0133	0.0007	0.0024	0.0020
Loss on ignition <sup>d</sup>	%	17.14	0.19	0.90	0.61
Mo <sup>e, f</sup>	µg/g	16.1	0.7	3.1	1.9
Moisture <sup>g, f</sup>	%	0.150	0.013	0.034	0.024
Na <sup>b</sup>	%	0.0147	0.0009	0.0009	0.0006
Ni	µg/g	7.27	0.43	0.81	0.64
Se	µg/g	304	8	40	18
Ti	µg/g	54.9	1.9	5.4	4.6

*a fusion followed by ion selective electrode was used exclusively by the laboratories*

*b statistical analysis of the data warrants classification as provisional despite only 6 sets of data*

*c data by flame atomic absorption spectrometry was included only, based on statistical tests*

*d samples of 1 to 2 grams ignited for 1 to 5 hours at 705 to 1050°C*

*e there is some evidence that, under certain conditions, digestion using nitric and hydrochloric acids, is incomplete*

*f 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*

*g samples of 1 to 5 grams dried for 1 to 5 hours at 105 to 110°C*

**Table 3 – CCU-1e Indicative Values (semi-quantitative only)**

Element	Units	Mean	No. accepted laboratories / values	Element	Units	Mean	No. accepted laboratories / values
Al AD2 <sup>a</sup>	%	0.1	4 / 20	Nd	µg/g	1	3 / 15
Ba	µg/g	6	4 / 20	Rb	µg/g	0.5	6 / 30
Bi	µg/g	3	8 / 40	Re	µg/g	0.004	4 / 20
Ce	µg/g	2	6 / 30	Sc	µg/g	1	7 / 35
Co AD2 <sup>a</sup>	µg/g	270	8 / 38	Sm	µg/g	0.3	3 / 15
Fe ICPE <sup>b</sup>	%	30.3	19 / 93	Sn	µg/g	15	7 / 34
Fe TITN <sup>c</sup>	%	31.2	6 / 30	Sr	µg/g	2	8 / 40
Ga	µg/g	3	7 / 35	Th	µg/g	0.2	3 / 15
Ge	µg/g	1	3 / 15	U	µg/g	1	6 / 30
In	µg/g	6	5 / 25	V	µg/g	4	7 / 35
La	µg/g	1	6 / 29	W	µg/g	0.4	3 / 15
Li	µg/g	0.5	4 / 20	Y	µg/g	1	6 / 30
Mn AD2 <sup>a</sup>	µg/g	80	4 / 20	Zr	µg/g	5	5 / 25
Nb	µg/g	0.2	5 / 25				

*a data by digestion using hydrochloric and nitric acids only was included*

*b data by inductively coupled plasma – optical emission spectroscopy only was included*

*c data by titration only was included*

#### **SOURCE**

CCU-1e 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-1e was obtained from the same company as its predecessor, CCU-1d.

#### **DESCRIPTION**

The mineral species include: chalcopyrite (63.2%); pyrite (21.6%); sphalerite (4.6%); mawsonite (2.8%); pyrrhotite and pyroxene (both at 2.7%); galena (0.6%); fergusonite (0.4%); chlorite (0.3%); arsenopyrite, magnetite and quartz (all at 0.2%); ankerite, calcite and hornblende (all at 0.1%); biotite (0.05%); plagioclase (0.04%); dolomite and riebeckite (both at 0.03%); albite, epidote, hematite and siderite (all at 0.02%); and allanite, annite, ilmenite, orthoclase and an unidentified mineral (all at 0.01%).

#### **INTENDED USE**

CCU-1e 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-1e 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 25°C, sieved, mixed and put into bottles each containing 200 grams. The recovery was 90% 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 1 gram was analyzed using electrodeposition with correction for co-plated impurities and residual copper in solution. Gold in samples of 5 grams was concentrated using lead fire assay and determined using gravimetric analysis.

Three subsamples for each bottle in a second set of 15 randomly chosen bottles were analyzed. Samples of 0.05 grams were digested using hydrochloric, nitric, hydrofluoric and boric acids. Aluminum, cobalt, manganese, lead and zinc were analyzed by inductively coupled plasma – atomic emission spectrometry. Antimony was analyzed by inductively coupled plasma – mass spectrometry.

The evidence indicates that CCU-1e is sufficiently homogeneous for use as a certified reference material. Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters.

## **CERTIFIED VALUES**

Thirty 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 using a hot plate or microwave oven, and various separation techniques followed by classical measurement methods such as iodometric and various titration methods, ISO 10258:1994, electrogravimetric analysis, gravimetric methods, ISO 10469:2006; and also instrumental techniques 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 other types of fire assay pre-concentration followed by atomic absorption spectrometry, gravimetric analysis, inductively coupled plasma – optical emission spectrometry and inductively coupled plasma – mass spectrometry were also used.

The methods used for various other elements included multi-acid digestions using a hot plate or microwave oven, and 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. In addition to these methods, cold vapour atomic absorption spectrometry was used for the determination of mercury. Various types of fusions, fused pellet and pressed powder pellet were followed by X-ray fluorescence. Instrumental neutron activation analysis was also used.

Sulphur and silicon were determined by gravimetric analysis. Carbon and sulphur were determined by combustion infrared spectroscopy. Various fusions and ion specific electrode were used for the determination of fluorine.

A one-way analysis technique (ANOVA<sup>1</sup>) 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. Twenty-two means were certified (see Table 1). Three 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 33:2015, section 8 - 9, and the publication, "Assessment of Laboratory Performance with CCRMP Certified Reference Materials", at [www.ccrmp.ca](http://www.ccrmp.ca).

#### **UNCERTIFIED VALUES**

Ten 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 iron by atomic absorption spectroscopy and sodium. Informational values for 27 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-1e 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**

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 OFFICER**



Maureen E. Leaver – CCRMP Coordinator

#### **FOR FURTHER INFORMATION**

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

#### **REFERENCES**

1. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960.