

CCRMP

Canadian Certified Reference Materials Project

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PCMRC

Projet canadien de matériaux de référence certifiés

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Certificate of Analysis

First issued: November 2000

Updated: January 2006

CCU-1c

Copper Concentrate Certified Reference Material

Element	Unit	Mean	Within-Lab Standard Deviation	Between- Lab Standard Deviation	95% confidence limit
Arsenic	µg/g	34	4	9	± 6
Cadmium	µg/g	136	5	12	± 6
Calcium Oxide	%	0.15	0.02	0.02	± 0.01
Carbon	%	0.09	0.01	0.02	± 0.01
Copper	%	25.62	0.07	0.12	± 0.05
Gold	µg/g	4.94	0.29	0.22	± 0.13
Iron	%	29.34	0.48	.68	± 0.28
Magnesium Oxide	%	1.02	0.04	0.06	± 0.04
Manganese	%	0.012	0.002	0.002	± 0.001
Molybdenum	µg/g	20	2	5	± 4
Selenium	µg/g	107	16	23	±15
Silicon Dioxide	%	2.52	0.07	0.16	± 0.10
Silver	µg/g	129	2	5	± 2
Sulphur	%	33.3	0.2	0.5	± 0.3
Zinc	%	3.99	0.06	0.19	± 0.07

Table 1 - CCU-1c Certified Values

SOURCE

Canada

CCU-1c is a copper flotation concentrate prepared from ore from the Ruttan mine, Lynn Lake, Manitoba, and donated by Hudson Bay Mining and Smelting, Flin Flon, Manitoba, Canada.



Canada

DESCRIPTION

Copper concentrates generally contain chalcopyrite, pyrite, sphalerite, pyrrhotite and gangue minerals. CCU-1c is the fourth in a series, all from the same source, with predecessors, CCU-1b, CCU-1a and CCU-1. The first three materials in the series are no longer available.

INTENDED USE

CCU-1c is suitable for the analysis of copper, and other elements at major, minor and trace levels in materials with a similar matrix. Examples of intended use are for quality control, method development, arbitration and the calibration of equipment.

INSTRUCTIONS FOR USE

The assigned values pertain to the date when issued. CANMET is not responsible for changes occurring after receipt by the user. CCU-1c should be analyzed "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.

HAZARDOUS SITUATION

Normal safety precautions, such as the use of safety glasses, breathing protection for fine particulate matter, gloves and a laboratory coat are suggested.

METHOD FOR PREPARATION

The raw material was dried at 66°C, sieved and blended to obtain a sub-74 micron (-200 mesh) product, which was bottled in 200-gram units. This is the only size available. Each bottle was sealed under nitrogen in a laminated aluminum foil-mylar pouch to prevent oxidation.

LEVEL OF HOMOGENEITY

The homogeneity of the stock with respect to its antimony, copper, gold, selenium, silver and zinc was investigated using twenty-two bottles chosen according to a stratified random sampling scheme. Two splits were analyzed from each bottle. The analyses for copper were performed by CANMET on 0.5 g samples using a multi-acid digestion followed by a titration with thiosulphate. Instrumental neutron activation was used on 0.5 g sample for antimony, selenium, silver and zinc and on a 5 g sample for gold. A one–way analysis of variance technique (ANOVA) was used to assess the homogeneity of these elements (1). The ratio of the between-bottles to within-bottle mean squares is compared to the F statistic at the 95% level of probability. No evidence of inhomogeneity was observed for all six elements. Use of a smaller sub-sample will invalidate the use of the certified value and associated parameters. Further details are available in the certification report.

CERTIFICATION

Twenty-eight industrial, commercial and government laboratories participated in an interlaboratory certification program. Various elements were analyzed by methods of each laboratory's choice. Depending on the element, the separation step was performed using multi-acid digestion, microwave digestion, fusions, and solvent extraction. Determinations were performed using colourimetric analysis, cold vapour atomic absorption spectroscopy for mercury, direct current arc, direct current plasma, electrogravimetry, electrolysis, atomic absorption, graphite furnace atomic absorption spectroscopy, gravimetry, hydride generation atomic absorption spectroscopy, high pressure liquid chromatography, inductively coupled plasma – atomic emission spectroscopy, inductively coupled plasma – mass spectroscopy, ion specific electrode, combustion, titrimetry and x-ray fluorescence.

A one-way analysis of variance technique was used to estimate the consensus value and other statistical parameters (1). Fifteen elements were assigned certified values based on statistical criteria. These are indicated in Table 1.

UNCERTIFED VALUES

Seven elements were assigned provisional values based on statistical criteria. These are indicated in Table 2. Informational values for fourteen elements were derived from the mean of fifteen to forty-seven results from a varying number of laboratories.

Element	Unit	Mean	Within Lab Standard Deviation	Between Lab Standard Deviation	95% confidence limit
Aluminum Oxide	%	0.34	0.03	0.11	± 0.07
Bismuth	µg/g	70	5	9	± 8
Chromium	µg/g	30	3	9	± 8
Cobalt	µg/g	18	1	5	± 3
Lead	%	0.34	0.01	0.02	± 0.01
Mercury	µg/g	32	2	8	± 5
Nickel	µg/g	11	2	4	± 4

Table 2 - CCU-1c Provisional Values

Table 3 - CCU-1c Informational Values

Element	Unit	Mean	Range	No. of Values
Antimony	µg/g	4	4-7	47
Barium	µg/g	31	6 - 76	21
Chlorine	µg/g	40	26 - 75	15
Fluorine	µg/g	294	190 - 368	30
Loss on ignition	%	16.4	15.8 - 16.8	15
Potassium	%	0.03	0.01 - 0.07	13
Scandium	µg/g	2	0.4 - 4	22
Sodium	%	0.02	0.007 - 0.08	37
Strontium	µg/g	4	3 - 5	20
Tellurium	µg/g	23	16 - 28	35
Titanium	µg/g	64	37 - 100	30
Vanadium	µg/g	15	2 - 34	20
Yttrium	µg/g	2	0.9 - 2	10
Zirconium	µg/g	17	6 - 23	15

TRACEABILITY

The certified values quoted herein are based on the consensus values derived from the statistical analysis of the data from the interlaboratory measurement program.

DATE OF CERTIFICATION

CCU-1c was originally released in November 2000. The second version of the certificate was issued in January 2004 due to the addition of an informational value for titanium, and the removal of an informational value for thallium. This version of the certificate, which was issued due the expiration of the second version, contains no changes in the values. Additional information in the text is included in conformance with ISO Guide 31:2000.

PERIOD OF VALIDITY

These certified values are valid until 2011. The stability of the material will be monitored every two years. Purchasers will be notified of any significant changes.

LEGAL NOTICE

CANMET has prepared this reference material and statistically evaluated the analytical data of the interlaboratory certification program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies CANMET 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 preparation and certification procedures used for CCU-1c, including methods and values obtained by individual laboratories, are given in CCRMP Report 00-3E. This report is available free of charge on application to:

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REFERENCE

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