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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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RTS-5

Certified Reference Material for a Nickel-Copper-Gold Tailings

Table 1 – RTS-5 Certified Values

note: For some elements, some methods do not produce a complete recovery. Where possible, certified, provisional or informational values are provided for those cases.

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	1.50	0.07	0.10	0.04
Al (FUS) [a]	%	6.25	0.04	0.13	0.08
As	µg/g	1286	27	84	28
Au (no AD2) [b]	µg/g	0.408	0.017	0.017	0.007
Ba (AD3, AD4, FUS, PPP) [c]	µg/g	252	5	11	5
Ca (AD3, AD4, FUS) [d]	%	3.86	0.06	0.16	0.06
Co (AD2) [e]	µg/g	69.8	1.4	4.2	3.0
Co (AD3, AD4, FUS, PPP) [c]	µg/g	76.9	1.5	3.9	1.7
Cr (T) [g]	µg/g	261	8	16	11
Cu	µg/g	647	12	25	8
Fe (AD4, FUS) [h]	%	11.9	0.14	0.38	0.15
K (AD3, AD4, FUS) [d]	%	0.850	0.013	0.055	0.023
Mg (AD3, AD4) [i]	%	3.31	0.06	0.15	0.09
Mg (FUS) [a]	%	3.59	0.03	0.11	0.07
Mn (AD3, AD4, FUS, PPP) [c]	µg/g	1092	19	40	16

cont'd

Table 1 – RTS-5 Certified Values *cont'd*

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Na (AD3, AD4, FUS) [d]	%	1.285	0.023	0.064	0.031
Ni (AD2) [e]	µg/g	1023	17	46	33
Ni (T) [f]	µg/g	1104	25	59	24
P	%	0.0369	0.0012	0.0040	0.0015
Pb (AD2, AD3) [j]	µg/g	57.6	4.0	5.7	3.2
Pb (AD4, FUS, PPP) [f]	µg/g	66.3	2.4	5.9	2.7
S	%	1.924	0.031	0.067	0.023
Si [a]	%	19.20	0.21	0.51	0.25
Sr (AD3, AD4, FUS, PPP) [c]	µg/g	130.6	2.3	7.2	3.3
Ti (T) [k]	%	0.3132	0.0035	0.0059	0.0037
Zn (AD2) [e]	µg/g	76.6	1.9	6.9	5.0
Zn (AD3, AD4, FUS, PPP) [c]	µg/g	105	4	10	4

a the data was obtained from various types of fusion based on statistical tests

b the data was obtained from fire assay pre-concentration only based on statistical tests

c the data was obtained from digestion with three acids (hydrochloric, nitric and hydrofluoric), four acids (hydrochloric, nitric, hydrofluoric and perchloric), various fusions and pressed powder pellet followed by x-ray fluorescence based on statistical tests

d the data was obtained from digestion with three acids, four acids and various fusions based on statistical tests

e the data was obtained from digestion with two acids (hydrochloric and nitric) based on statistical tests

f the data was obtained from digestion with four acids, various fusions and pressed powder pellet followed by x-ray fluorescence based on statistical tests

g the data was obtained from digestion with four acids in a closed vessel, various fusions, pressed powder pellet followed by x-ray fluorescence and instrumental neutron activation analysis based on statistical tests

h the data was obtained from digestion using four acids and various fusions based on statistical tests

i the data was obtained from digestion with three acids and four acids based on statistical tests

j the data was obtained from digestion with two and three acids based on statistical tests

k the data was obtained from digestion with four acids in a closed vessel and various fusions based on statistical tests

Table 2 – RTS-5 Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al (AD3, AD4) [a]	%	5.66	0.10	0.44	0.26
Ba (AD2) [b, c]	µg/g	127.1	2.0	5.8	5.4
Bi (AD) [d]	µg/g	2.05	0.07	0.23	0.19
C [e]	%	1.617	0.013	0.035	0.030
Ca (AD2) [b]	%	2.83	0.03	0.21	0.17
Ce [c]	µg/g	17.0	1.0	1.9	1.8
Cr (AD3, AD4) [a]	µg/g	216	4	31	17
K (AD2) [b, c]	%	0.352	0.004	0.011	0.012
La	µg/g	9.7	0.5	1.8	1.1
Li [c, d]	µg/g	16.9	0.5	1.2	1.1
Loss on ignition [f]	%	9.90	0.13	0.82	0.55
Mo [c]	µg/g	1.338	0.055	0.086	0.083
Rb [c]	µg/g	30.5	0.8	1.8	2.0
S (SO_4)	%	1.23	0.02	0.14	0.08
Se (AD) [d, g]	µg/g	8.03	0.55	0.82	0.58
Sr (AD2) [b]	µg/g	41.8	0.8	2.4	2.0
Th [c]	µg/g	2.25	0.28	0.35	0.34
Ti (AD3, AD4) [a]	%	0.245	0.005	0.034	0.018
V (AD2) [b]	µg/g	61.2	1.3	4.1	3.4
V (AD3, AD4) [a]	µg/g	136	3	20	10
V (T) [h]	µg/g	160	3	18	15
Y (AD3, AD4) [i]	µg/g	10.1	0.3	1.4	1.2
Y (T) [c, h]	µg/g	15.5	0.7	1.2	1.3

a the data was obtained from digestion using three (hydrochloric, nitric and hydrofluoric) and four acids (hydrochloric, nitric, hydrofluoric and perchloric) based on statistical tests
b the data was obtained from digestion with two acids (hydrochloric and nitric) based on statistical tests
c statistical analysis of the data warrants classification as provisional despite only 6 or 7 sets of data
d the data was obtained from digestion with various mixtures of acids
e the data was obtained from a combustion apparatus with detection by infrared spectroscopy
f the data is based on samples of 0.2 to 2 grams ignited for 0.5 to 5 hours at 950 to 1050°C
g the data fulfilled the conditions for certification but the element was reclassified as provisional since much of the data consisted of one significant figure
h the data was obtained from digestion using four acids in closed vessel, fusion and pressed powder pellet followed by x-ray fluorescence based on statistical tests
i the data was obtained from digestion using three and four acids based on statistical tests

Table 3 – RTS-5 Informational Values (semi-quantitative only) *cont'd*

Analyte	Units	Mean	No. accepted laboratories / values	Analyte	Units	Mean	No. accepted laboratories / values
Al (AD2) [a]	%	1.6	7 / 35	S elemental	%	0.3	3 / 15
Be	µg/g	0.7	9 / 45	Sb	µg/g	1.3	5 / 25
Cr (AD2) [a]	µg/g	95	7 / 35	Sc (AD2) [a]	µg/g	9	4 / 20
Cs (AD) [b]	µg/g	1.0	5 / 25	Sc (AD3) [f]	µg/g	25	3 / 15
Dy (AD) [b]	µg/g	2	4 / 20	Sc (AD4) [g]	µg/g	30	7 / 35
Er (AD) [b]	µg/g	2	4 / 20	Sc (T) [h]	µg/g	35	6 / 30
Eu (AD) [b]	µg/g	0.6	4 / 20	Sm [b]	µg/g	2	4 / 20
Fe (AD2) [a]	%	10	7 / 35	Sn [b]	µg/g	3	6 / 30
Fe (AD3) [c]	%	11	5 / 25	Ta [b]	µg/g	0.3	4 / 20
Ga	µg/g	14	14 / 70	Tb [b]	µg/g	0.3	3 / 15
Lu	µg/g	0.3	4 / 20	Te (AD) [b, i]	µg/g	1.2	6 / 30
Mg (AD2) [a]	%	2.2	6 / 30	Ti (AD2) [a]	%	0.07	5 / 25
Moisture [d]	%	1.4	10 / 50	Tl [b]	µg/g	0.3	5 / 25
Mn (AD2) [a]	µg/g	750	7 / 35	U [b, i]	µg/g	0.5	5 / 25
Na (AD2) [a]	%	0.08	5 / 25	Y (AD2) [a]	µg/g	3	4 / 20
Nb	µg/g	4	5 / 25	Yb	µg/g	2	4 / 20
Nd	µg/g	8	5 / 25	Zr (AD2) [c]	µg/g	4	5 / 25
Pd [e]	µg/g	0.14	4 / 20	Zr (AD3, AD4) [j]	µg/g	49	8 / 40
Pt [e]	µg/g	0.2	4 / 20	Zr (T) [k]	µg/g	75	6 / 30

a the data was obtained from digestion with two acids (hydrochloric and nitric)
b the data was obtained from various acid digestion methods only
c the data was obtained from digestion using three acids (hydrochloric, nitric and hydrofluoric)
d the data was obtained from samples of 1 to 10 grams dried for 0.5 to 20 hours at 80 to 105°C; the data fulfills the conditions for provisional but the parameter was reclassified as informational due to the between-laboratory standard deviation being 24% of the mean
e the data was obtained by lead fire assay using a test portion of 25-30 grams
f the data was obtained from digestion using three acids
g the data was obtained from digestion using four acids (hydrochloric, nitric, hydrofluoric and perchloric)
h the data was obtained from digestion using four acids in a closed vessel, fusion, pressed powder pellet followed by x-ray fluorescence and instrumental neutron activation analysis
i only data obtained from inductively coupled plasma - mass spectrometry was used
j the data was obtained from digestion using three and four acids
k the data was obtained from digestion using four acids in closed vessel, fusion and pressed powder pellet followed by x-ray fluorescence

SOURCE

RTS-5 is a nickel-copper-gold tailings prepared from a mixture of tailings from two Canadian mining companies.

DESCRIPTION

The mineral species include: quartz (17.1%); hornblende (8.5%); chamosite (+Mg) (8.2%); albite (8.1%); dolomite (7.5%); oligoclase (6.5%); clinochlore (+Fe) (6.0%); kaolinite (5.7%); muscovite (4.0%); biotite (3.5%); ferroan-hornblende (2.8%); limonite and garnet (each at 2.5%); yavapaiite (2.2%); goethite (1.8%); miscellaneous silicates (1.7%); olivine and ferroan-dolomite (+Mn) (each at 1.5%); ferroan-magnesite (+Mn) (1.1%); pyrite (1.0%); ferroan-dolomite and pyrrhotite (each at 0.8%); K-feldspar (0.7%); plagioclase (0.6%); hematite (0.5%); eulite (0.4%); ilmenite and other oxides (each at 0.3%); magnetite, cummingtonite, magnesio-siderite, chalcopyrite, apatite and arsenopyrite (each at 0.2%); titanomagnetite, rutile, titanite and vesuvianite (each at 0.1%), ankerite and calcite (each at 0.04%); clay (Mn rich), siderite, magnesio-siderite (+Ca) and andradite (each at 0.03%); manganoan-calcite and miscellaneous (each at 0.02%); and grunerite, zircon, siderite (+Mn), rhodochrosite, magnesio-siderite (+Mn), and chromite (each at 0.01%).

INTENDED USE

RTS-5 is suitable for the analysis of nickel, copper, gold and other elements in tailings in concentrations ranging from major, minor to trace levels. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE

RTS-5 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 32°C. After crushing and sieving, the recovery of the minus 75 µm fraction was 702%. The product was blended, and then bottled in 100-gram units. Each bottle was purged with nitrogen and sealed in a laminated polyethylene - foil pouch to prevent oxidation. This is the only size that is available.

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. Subsamples of 0.25 grams were digested using hydrochloric, nitric, hydrofluoric and perchloric acids, and analyzed by inductively coupled plasma – optical emission spectrometry for arsenic, cobalt, copper and nickel. Samples of 0.15 grams were analyzed for sulphur using a combustion apparatus with infrared detection.

Additionally, a set of 15 pairs of bottles was chosen according to a stratified random sampling scheme. Each pair of consecutively numbered bottles was combined to form a 200 gram sample. Three subsamples were selected from each of the combined bottles i.e. 200 grams. Gold was determined in subsamples of 30 grams using fire assay pre-concentration followed by determination using inductively coupled plasma – mass spectrometry.

Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters. A one–way analysis of variance technique (ANOVA)[1] and statistical analyses were used to assess the homogeneity of these elements. No significant between-bottle variation was observed for all elements.

CERTIFIED VALUES

Twenty-nine industrial, commercial and government laboratories participated in an interlaboratory measurement program using methods of their own choice.

Methods for the determination of the elements included digestion with various combinations of acids; and various types of fusions followed by the determination using atomic absorption spectrometry, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry. X-ray fluorescence on a pressed powder pellet and instrumental neutron activation were also used for many elements.

The concentration of carbon was determined using combustion followed by infrared spectrometry.

The concentration of sulphur was determined using combustion followed by infrared spectrometry, gravimetric analysis, various acid digestions and fusions followed by inductively coupled plasma – optical emission spectroscopy and X-ray fluorescence. The concentration of sulphur species was determined using the same methods as sulphur, plus leaches followed by inductively coupled plasma – optical emission spectroscopy or high pressure liquid chromatography.

Gold was determined by fire assay pre-concentration using lead button collection, followed by determination using atomic absorption spectroscopy, inductively coupled plasma – optical emission spectrometry and inductively coupled plasma – mass spectrometry.

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's statistical criterion regarding the agreement of the results. Twenty-seven analytes 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.cccrmp.ca.

UNCERTIFIED VALUES

Twenty-three provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CCRMP 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 both barium and potassium by digestion using two acids; and cerium, lithium, molybdenum, rubidium, thorium and yttrium. Informational values for thirty-eight analytes, 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

RTS-5 was released as a new material in April 2015.

PERIOD OF VALIDITY

The certified values are valid until April 30, 2035. The stability of the material will be monitored every two years for the duration of the inventory. Updates, if needed, 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.

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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.