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CCRMP
Canadian Certified Reference Materials Project

PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: September 2010 Version: September 2010

GTS-2a

Certified Reference Material for a Gold Ore Mill Tailings

Table 1 – GTS-2a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al (no AD2)[a]	%	6.96	0.11	0.22	0.12
As	µg/g	124	4	11	5
Au (FA)[b]	µg/g	0.272	0.009	0.011	0.005
Ba (no AD2)[a]	µg/g	186	8	14	7
C	%	2.011	0.021	0.063	0.041
Ca	%	4.01	0.09	0.14	0.06
Co	µg/g	22.1	0.8	1.7	0.7
Cu	µg/g	88.6	2.9	6.1	2.5
Fe	%	7.56	0.13	0.26	0.11
K (no AD2)[a]	%	2.021	0.036	0.064	0.035
Mg (no AD2)[a]	%	2.412	0.038	0.075	0.038
Mn	µg/g	1510	30	120	50
Na (no AD2)[a]	%	0.617	0.016	0.063	0.033
Ni	µg/g	77.1	2.9	9.1	3.7
P	%	0.0892	0.0030	0.0084	0.0036
S	%	0.348	0.009	0.028	0.014
Si	%	23.65	0.14	0.21	0.16
Sr (no AD2)[a]	µg/g	92.8	2.2	6.0	3.2
Th (no AD2)[a]	µg/g	1.244	0.125	0.090	0.076
Zn	µg/g	208	7	21	9

a sets with digestions by two acids, usually hydrochloric and nitric acids, were excluded as method outliers based on statistical tests

b fire assay techniques only i.e. sets by digestion excluded as statistical outliers

CANMET Mining and Mineral Sciences Laboratories
555 Booth Street, Ottawa, Ontario, Canada K1A 0G1
Tel.: (613) 995-4738, Fax: (613) 943-0573
E-mail: ccrmp@nrcan.gc.ca
www.ccrmp.ca

Laboratoires des mines et des sciences minérales de CANMET
555, rue Booth, Ottawa (Ontario) Canada K1A 0G1
Tél. : (613) 995-4738, Téléc. : (613) 943-0573
Courriel : pcmrc@rncan.gc.ca
www.pcmrc.ca

Canada

Table 2 – GTS-2a Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al (AD2)[a,b]	%	1.80	0.07	0.12	0.13
Be (no AD2)[a,c]	µg/g	0.882	0.049	0.044	0.052
Cd[e]	µg/g	0.58	0.10	0.21	0.12
Ce (no AD2)[c]	µg/g	24.35	0.86	0.50	0.49
Ga (no AD2)[c]	µg/g	21.1	0.7	3.6	2.6
Hg	µg/g	0.220	0.028	0.047	0.036
La (no AD2)	µg/g	9.44	0.31	0.62	0.49
LOI[f]	%	9.87	0.04	0.25	0.19
Mg (AD2)[a,b]	%	2.173	0.048	0.095	0.102
Mo	µg/g	3.84	0.27	0.21	0.21
Nd (no AD2)[a,c]	µg/g	16.57	0.56	0.71	0.79
Pb	µg/g	17.9	1.4	5.7	2.6
Pr[a]	µg/g	3.45	0.12	0.18	0.20
Rb (no AD2)[c]	µg/g	57.7	1.5	2.3	2.0
Sb (no AD2)[c]	µg/g	1.33	0.13	0.13	0.11
Sc (no AD2)[c]	µg/g	29.3	0.7	3.0	2.0
Sm	µg/g	4.58	0.11	0.44	0.37
Sr (AD2)[a,b]	µg/g	63.2	1.5	1.8	2.0
Te	µg/g	1.64	0.30	0.46	0.30
Ti (FUS)[d]	µg/g	0.500	0.006	0.013	0.010
V (no AD2)[c]	µg/g	166	6	30	17
Yb (T)[a,g]	µg/g	4.41	0.12	0.21	0.23
W (no AD2)[c]	µg/g	25.8	1.2	6.7	4.5
Zr (no AD2)[c]	µg/g	114	6	26	15

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

b includes digestion by two acids only, usually hydrochloric and nitric

c sets by digestion by two acids, usually hydrochloric and nitric acids, excluded as method outliers based on statistical tests

d includes fusions only

e data fulfilled the conditions for certification, but the element was re-classified as provisional since much of the data had one significant figure

f loss on ignition for a sample of 1-2 g for 0.25 - 3 hour at a temperature of 900 to 1050°C

g includes only total recovery (T) methods such as digestion by four acids in a closed beaker, various fusions and instrumental neutron activation analyses; the exclusion of sets by acid digestions in an open beaker as method outliers was based on statistical tests

Table 3 – GTS-2a Informational Values

Element	Units	Mean	No. accepted laboratories / values	Element	Units	Mean	No. accepted laboratories / values
Ag	µg/g	0.64	11 / 51	Na (AD2)[b]	%	0.015	5 / 26
Au (AD2,4)[a]	µg/g	0.25	3 / 14	Nb (no AD2)[d]	µg/g	4	7 / 34
Bi	µg/g	0.3	7 / 34	Pd	µg/g	0.002	3 / 15
Cr (AD2)[b]	µg/g	140	7 / 37	Sn	µg/g	1	7 / 34
Cr (T)[c]	µg/g	270	5 / 25	Ta	µg/g	0.3	3 / 15
Cs (no AD2)[d]	µg/g	1.7	5 / 24	Tb (T)[c]	µg/g	1.1	5 / 25
Dy (T)[c]	µg/g	7	4 / 20	Ti (AD3,4)	%	0.3	7 / 34
Er (T)[c]	µg/g	4.4	4 / 20	Tl (AD4)	µg/g	0.40	5 / 24
Eu (T)[c]	µg/g	1.5	5 / 25	Tm (T)[c]	µg/g	0.66	4 / 20
Gd (T)[c]	µg/g	6.3	4 / 20	U	µg/g	0.4	3 / 15
Hf (T)[c]	µg/g	3.5	6 / 30	V (AD2)[b]	µg/g	50	4 / 20
Ho (T)[c]	µg/g	1.5	4 / 20	W (AD2)[b]	µg/g	12	4 / 20
Li	µg/g	27	7 / 34	Y (T)[c]	µg/g	38	6 / 30
Lu (T)[c]	µg/g	0.7	6 / 30	Zr (AD2)[b]	µg/g	5	4 / 20

a includes digestion by two acids, usually hydrochloric and nitric acids, and four acids only

b includes digestion by two acids, usually hydrochloric and nitric acids

c includes only total recovery (T) methods such as digestion by four acids in a closed vessel, various fusions and instrumental neutron activation analyses; the exclusion of sets by acid digestions in an open beaker as method outliers was based on statistical tests

d digestion by two acids, usually hydrochloric and nitric acids, was excluded as a method outlier based on statistical tests

SOURCE

GTS-2a is a gold ore mill tailings obtained from a tailings dam at the Porcupine gold mine in Timmins, Ontario, Canada. The raw material was donated by Goldcorp Canada Limited, Porcupine Gold Mine. The raw material for GTS-2a was obtained from the same source as its predecessor, GTS-2.

DESCRIPTION

The mineral species include: quartz (33.5%); clinochlore (14.0%); K-feldspar (13.5%); ankerite (13.5%); muscovite (11.4%); albite (4.3%); calcite (2.1%); siderite (1.7%); biotite (1.2%); rutile and magnetite (both at 0.7%); pyrite and anorthite (both at 0.6%); various other trace minerals including several silicates, rare earths, arsenopyrite, sphalerite, chalcopyrite, melonite, altaite, graphite and gold (for a total of 0.6%); apatite (0.5%); pyrrhotite and Mg-ferrite (0.3%); talc, ilmenite and gypsum (all at 0.1%); and epidote (0.01%).

INTENDED USE

GTS-2a is suitable for the analysis of various elements at major, minor and trace levels in tailings. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE
GTS-2a should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. 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 32°C, crushed, sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction was 78%. The product was blended, and then bottled in 350-gram units.

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. The gold in 30-g subsamples was concentrated using lead fire assay and analyzed using inductively coupled plasma – mass spectrometry. Each bottle in a second set of 15 randomly chosen bottles was subsamples into 8 subsamples and three subsamples were analyzed. Subsamples of 0.25g grams were digested using four acids, hydrofluoric, hydrochloric, nitric and perchloric acids, and analyzed for copper and nickel by inductively coupled plasma – atomic emission spectrometry and for lead by inductively coupled plasma – mass spectrometry. Subsamples of 0.15 grams from each of the 3 subsamples per bottle were analyzed for sulphur using a combustion apparatus with infrared detection. 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] was used to assess the homogeneity of these elements. No significant between-bottle variation was observed for copper, gold, nickel, lead and sulphur.

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

Methods for the analysis of gold included pre-concentration by lead fire assay, digestion by two and four acids followed by determination using flame atomic absorption spectrometry, inductively coupled plasma – optical emission spectroscopy, inductively coupled plasma – mass spectrometry, gravimetric analysis and instrumental neutron activation analysis.

The methods used for various other elements included multi-acid digestions in open or closed vessel, microwave digestion, various types of fusions, and followed by determination by flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy, inductively coupled plasma – mass spectrometry. Various types of fusions followed by X-ray fluorescence, as well as instrumental neutron activation analysis were used to determine several elements. Hydride generation atomic absorption spectrometry was used for the determination of arsenic and tellurium. Loss on ignition was determined using sample weights of 1-2 grams for 0.25 - 3 hour at a temperature of 900 to 1050°C. Sulphur was determined by various acid digestions followed by inductively coupled plasma – optical emission spectrometry or gravimetric finish; preparation of a fused pellet followed by X-ray fluorescence; as well as combustion with infrared spectrometry. Silicon was determined by various fusions or acids digestions followed by inductively coupled plasma – optical emission spectroscopy, gravimetric analysis, or fusion followed by X-ray fluorescence.

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. Twenty elements

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

Twenty-four 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 sets of data for which the statistical analysis of the data warranted provisional status. This latter group includes (i) aluminum, magnesium and strontium by digestion with two acids, (ii) beryllium and neodymium by all methods except digestion with two acids, (iii) ytterbium by total recovery methods and (iv) praseodymium by all methods. Informational values for 28 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

GTS-2a is a new material.

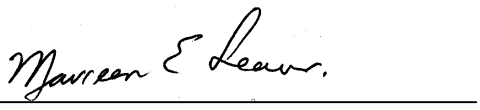
PERIOD OF VALIDITY

The certified values are valid until September 30, 2032. 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



Maureen E. Leaver – CCRMP Coordinator

Joseph Salley – Data Processor

FOR FURTHER INFORMATION

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

CCRMP
CANMET-MMSL (NRCan)
555 Booth Street, room 433
Ottawa, Ontario, Canada K1A 0G1

Telephone: (613) 995-4738
Facsimile: (613) 943-0573
E-mail: ccrmp@nrcan.gc.ca

REFERENCES

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