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CCRMP
Canadian Certified Reference Materials Project

PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: February 2011 Version: June 2011

WMG-1a

Certified Reference Material for a Mineralized Gabbro with Gold and Platinum Group Elements

Table 1 – WMG-1a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	3.03	0.14	0.20	0.10
Al no AD2[a]	%	4.75	0.06	0.12	0.05
As	µg/g	5.99	0.93	0.93	0.43
Ba no AD2[a]	µg/g	216	6	18	8
Ca no AD2[a]	%	10.06	0.16	0.39	0.17
Co	µg/g	191	5	16	6
Cr AD4[b]	µg/g	670	40	120	80
Cr T[c]	µg/g	804	18	47	31
Cu	µg/g	7120	120	300	120
Dy	µg/g	2.291	0.060	0.060	0.042
Fe no AD2,3[d]	%	12.71	0.20	0.56	0.23
K no AD2[a]	%	0.1021	0.0054	0.0069	0.0038
La no AD2[a]	µg/g	8.47	0.21	0.48	0.26
Mg no AD2[a]	%	7.41	0.10	0.26	0.11
Mo	µg/g	2.49	0.14	0.20	0.14
Na no AD2[a]	%	0.1119	0.0043	0.0090	0.0046
Nd	µg/g	9.41	0.17	0.48	0.32
Ni	µg/g	2480	50	100	40
P	%	0.0731	0.0032	0.0040	0.0019
Pd	µg/g	0.484	0.013	0.018	0.009

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Table 1 – WMG-1a Certified Values *cont'd*

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Pt	µg/g	0.899	0.021	0.036	0.018
S no AD [e]	%	3.43	0.04	0.11	0.07
Sc no AD2[a]	µg/g	21.33	0.51	1.28	0.78
Se	µg/g	14.1	0.6	1.4	0.9
Si	%	18.27	0.15	0.20	0.13
Sm	µg/g	2.211	0.062	0.062	0.040
Sr no AD2[a]	µg/g	39.0	1.0	3.4	1.7
Th	µg/g	1.07	0.05	0.11	0.06
Ti no AD2[a]	%	0.419	0.011	0.016	0.007
V no AD2[a]	µg/g	158	4	13	6
Y no AD2[a]	µg/g	12.67	0.37	0.85	0.50
Zn	µg/g	112	6	17	7
Zr AD4[b]	µg/g	35.7	1.0	2.3	1.5

a sets using digestion by two acids, usually hydrochloric and nitric acids, were excluded as method outliers based on statistical tests
b sets using digestion using four acids, usually hydrochloric, nitric, hydrofluoric and perchloric acids, were included only based on statistical tests
c sets by total recovery methods such as various fusions and instrumental neutron activation analyses were included only; the exclusion of sets by acid digestions as method outliers was based on statistical tests
d sets using digestion by two or three acids, usually hydrochloric, nitric without/with hydrofluoric acid, were excluded as method outliers based on statistical tests
e sets by combustion and fusion methods only were included

Table 2 – WMG-1a Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Au[a]	µg/g	0.0617	0.0117	0.0117	0.0039
Bi[b]	µg/g	0.251	0.019	0.019	0.012
Cd AD4[b, c]	µg/g	0.818	0.057	0.090	0.086
Ce no AD2[a,d]	µg/g	17.18	0.45	0.56	0.42
Er[b]	µg/g	1.34	0.040	0.086	0.081
Eu	µg/g	0.733	0.027	0.053	0.042

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Table 2 – WMG-1a Provisional Values *cont'd*

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ga	µg/g	12.4	0.5	3.5	1.9
Gd[b]	µg/g	2.351	0.066	0.075	0.085
Li	µg/g	44.7	1.4	7.2	4.2
Loss on ignition[e]	%	4.31	0.08	0.33	0.22
Lu[b]	µg/g	0.196	0.007	0.012	0.013
Mn no AD2,3[f]	µg/g	1141	21	88	37
Nb	µg/g	5.26	0.22	0.36	0.29
Pb[g]	µg/g	9.2	0.9	2.1	1.2
Pr[b]	µg/g	2.220	0.035	0.082	0.087
Rb no AD2[b, h]	µg/g	2.53	0.07	0.14	0.13
S AD [i]	%	3.03	0.08	0.33	0.24
Sb	µg/g	1.55	0.09	0.50	0.32
Sn	µg/g	1.91	0.38	0.63	0.50
Ta AD [b, i]	µg/g	0.355	0.032	0.054	0.058
Te	µg/g	1.19	0.11	0.28	0.23
Tm	µg/g	0.192	0.006	0.016	0.014
U T[b, j]	µg/g	0.65	0.033	0.072	0.068
Yb	µg/g	1.220	0.041	0.096	0.082

a data fulfilled the conditions for certification, but the element was re-classified as provisional since the within-laboratory standard deviation is 20% of the mean

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

c sets by digestion using four acids, usually hydrochloric, nitric, hydrofluoric and perchloric acids, were included only, based on statistical tests

d data fulfilled the conditions for certification, but the element was re-classified as provisional since 33% of the data were outliers

e samples of 0.5 to 2 grams dried at 950 to 1000°C for 0.5 to 12 hours included

f sets using digestion by two or three acids, usually hydrochloric, nitric without/with hydrofluoric acids, were excluded as method outliers based on statistical tests

g data fulfilled the conditions for certification but the element was reclassified as provisional since the between-laboratory standard deviation is 22% of the mean

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

i sets using acid digestions methods only included, based on statistical tests

j includes only total recovery methods such as various fusions and instrumental neutron activation analysis; the exclusion of sets by acid digestions as method outliers was based on statistical tests

Table 3 – WMG-1a Informational Values (semi-quantitative only)

Analyte	Units	Mean	No. accepted laboratories / values	Analyte	Units	Mean	No. accepted laboratories / values
Al AD2[a]	%	3.3	4 / 20	Mn AD2[a]	µg/g	350	5 / 25
Be	µg/g	0.4	4 / 20	moisture[e]	%	0.2	6 / 25
C	%	0.13	4 / 20	Na AD2[a]	%	0.02	4 / 18
Ca AD2[a]	%	2	5 / 20	Rh FA [f]	µg/g	0.015	4 / 20
Cd AD2[a]	µg/g	0.6	5 / 25	Ru FAN[g]	µg/g	0.02	3 / 15
Cr AD2[a]	µg/g	360	5 / 25	Sc AD2[a]	µg/g	2	3 / 15
Cs	µg/g	0.46	5 / 20	Sr AD2[a]	µg/g	19	6 / 30
Fe AD2[a]	%	9.5	4 / 20	Tb AD4 + FUS[h]	µg/g	0.40	5 / 25
Ge	µg/g	1	4 / 20	Ti AD2[a]	%	0.2	5 / 25
Hf AD4[b]	µg/g	1.1	3 / 15	Tl	µg/g	0.1	3 / 15
Hf T[c]	µg/g	1.4	4 / 20	U AD [i]	µg/g	0.44	7 / 35
Ho	µg/g	0.48	7 / 35	V AD2[a]	µg/g	54	5 / 25
In no AD2[d]	µg/g	0.2	4 / 18	W no AD2[d]	µg/g	0.6	4 / 20
Ir	µg/g	0.027	5 / 25	Y AD2[a]	µg/g	3.4	4 / 20
K AD2[a]	%	0.02	4 / 20	Zr AD2[a]	µg/g	7	5 / 25
La AD2[a]	µg/g	3.7	3 / 15	Zr FUS[j]	µg/g	50	5 / 25
Mg AD2[a]	%	3.5	5 / 25				

a sets by digestion using two acids only , usually hydrochloric and nitric acids, included
b sets by digestion using four acids only
c sets by total recovery methods such as various fusions and instrumental neutron activation analysis only included
d data suggests that digestions by two acids may have a lower recovery than other methods
e samples of 1-5 grams dried at 105°C for 3 minutes to 12 hours included
f sets by fire assay by either lead button or nickel sulphide collection only included
g sets by fire assay using nickel sulphide collection only included
h sets by digestion using four acids and fusions only included
i sets by digestion only included
j sets by fusion methods only included

SOURCE
WMG-1a is a mineralized gabbro with gold and platinum group elements obtained from the Wellgreen property in the Yukon, Canada. The raw material was donated by Northern Platinum Limited. The raw material for WMG-1a was obtained from the same source as its predecessor, WMG-1.

DESCRIPTION
The mineral species include: altaite (0.03%), amphibole (38.9%), apatite (0.4%), chalcopyrite (2.0%), clinochlore (10.0%), diopside (38.6%), ilmenite (0.1%), magnetite (0.4%), orthoclase (0.6%), otherite (0.1%), pentlandite (0.5%), pyrrhotite (6.9%) and titanite (1.6%).

INTENDED USE
WMG-1a is suitable for the analysis of gold, platinum and palladium and various elements at major, minor and trace levels in ores. Examples of intended use include quality control and method development. Caution is advised for the use of WMG-1a for the quality control of data for gold.

INSTRUCTIONS FOR USE
WMG-1a 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 32°C, crushed, ground, sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction was 71%. The product was blended, and then bottled in 350-gram units. 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. The gold, platinum and palladium in 15-g subsamples were 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 divided into 8 subsamples and three subsamples were analyzed. Subsamples of 0.25g grams were digested using hydrofluoric, hydrochloric, nitric and perchloric acids, and analyzed for aluminum, cobalt, copper and nickel by inductively coupled plasma – atomic 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 WMG-1a is sufficiently homogeneous for use as a certified reference material.

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

Methods for the analysis of gold, palladium and platinum included pre-concentration by fire assay using nickel sulphide or lead button collection, followed by determination using flame atomic absorption spectrometry, inductively coupled plasma – optical emission spectroscopy, inductively coupled plasma – mass spectrometry and instrumental neutron activation analysis.

The methods used for various other elements included multi-acid digestions, microwave digestion, various types of fusions, and followed by determination by flame atomic absorption spectroscopy, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry. Various types of fusions followed by X-ray fluorescence, as well as instrumental neutron activation analysis were also used. Hydride generation atomic absorption spectrometry was used for the determination of antimony, arsenic, selenium and tellurium.

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. Thirty-three parameters 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) bismuth by all methods and cadmium by digestion using four acids; (ii) erbium, gadolinium, lutetium, praseodymium and rubidium by all methods except digestion using two acids; (iii) tantalum by acid digestion methods only; and (iv) uranium by total methods such as fusions and instrumental neutron activation analysis. Informational values for 33 parameters, 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

WMG-1a was released as a new material in February 2011. In June 2011 the certificate was re-issued with the units for titanium in Tables 1 and 3 corrected from µg/g to %.

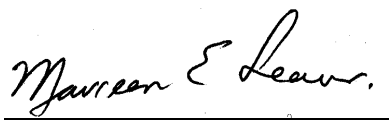
PERIOD OF VALIDITY

The certified values are valid until January 28, 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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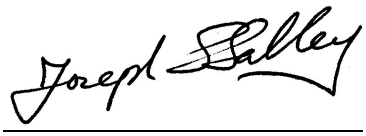

Joseph Salley – Data Processor

FOR FURTHER INFORMATION

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

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