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CCRMP
Canadian Certified Reference Materials Project

PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: April 2012 Version: April 2012

WPR-1a

Certified Reference Material for a Peridotite with Rare Earth and Platinum Group Elements

Table 1 – WPR-1a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	1.02	0.05	0.10	0.05
Al AD4 + no AD [a]	%	2.621	0.043	0.076	0.035
As	µg/g	9.3	0.7	1.1	0.6
Ba AD4 + no AD [a]	µg/g	70.6	2.3	2.4	1.4
Bi	µg/g	0.122	0.006	0.011	0.008
Ca no AD2 [b]	%	2.528	0.046	0.096	0.044
Cd	µg/g	0.598	0.037	0.086	0.044
Ce no AD2 [b]	µg/g	9.69	0.32	0.57	0.33
Co AD4 + no AD [a]	µg/g	213	4	17	8
Cs	µg/g	2.38	0.08	0.15	0.08
Cu	%	0.299	0.005	0.016	0.006
Dy no AD2 [c]	µg/g	1.624	0.056	0.056	0.035
Er AD4 + no AD [a]	µg/g	0.886	0.037	0.037	0.016
Eu AD4 + no AD [a]	µg/g	0.497	0.017	0.020	0.013
Fe AD4 + no AD [a]	%	11.34	0.20	0.49	0.20
Ga no AD2 [b]	µg/g	7.04	0.24	0.28	0.18
Gd no AD2 [c]	µg/g	1.76	0.05	0.10	0.06
Hf FUS [d]	µg/g	1.142	0.071	0.094	0.071
Ho AD4 + no AD [a]	µg/g	0.322	0.009	0.019	0.013
K	%	0.156	0.006	0.011	0.005

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

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
La AD4 + no AD [a]	µg/g	4.04	0.15	0.24	0.13
Li AD4 + no AD [a]	µg/g	25.6	0.8	1.1	0.8
Lu no AD2 [b]	µg/g	0.121	0.006	0.010	0.007
Mn no AD2 [b]	%	0.138	0.003	0.012	0.005
Nd no AD2 [c]	µg/g	6.26	0.20	0.20	0.10
Ni AD4 + no AD [a]	%	0.439	0.009	0.022	0.010
P no AD2 [c]	µg/g	303	19	19	9
Pb	µg/g	7.92	0.89	0.89	0.32
Pd	µg/g	0.614	0.015	0.025	0.012
Pr no AD2 [b]	µg/g	1.362	0.050	0.054	0.035
Pt	µg/g	0.452	0.014	0.030	0.014
Rb	µg/g	7.06	0.16	0.44	0.24
S [e]	%	1.768	0.033	0.079	0.038
Sb no AD2 [b]	µg/g	3.13	0.21	0.39	0.27
Sc AD4 + no AD [a]	µg/g	17.3	0.4	1.2	0.6
Si no AD2 [f]	%	17.62	0.17	0.34	0.18
Sm no AD2 [b]	µg/g	1.617	0.050	0.071	0.041
Sr AD4 + no AD [a]	µg/g	19.5	0.6	1.1	0.6
Tb no AD2 [c]	µg/g	0.269	0.010	0.020	0.013
Ti AD4 + no AD [a]	%	0.3527	0.0054	0.0082	0.0042
Tm AD4 + no AD [g]	µg/g	0.126	0.006	0.017	0.012
V AD4 + no AD [a]	µg/g	135	3	10	5
Y AD4 + no AD [a]	µg/g	8.39	0.23	0.43	0.26
Yb AD4 + no AD [a]	µg/g	0.790	0.027	0.052	0.034
Zn no AD2 [b]	µg/g	160	5	18	8

a sets using digestion by two acids (hydrochloric and nitric) and three acids (hydrochloric, nitric and hydrofluoric) were excluded as method outliers based on statistical tests

b sets using digestion by two acids (hydrochloric and nitric) were excluded as method outliers based on statistical tests

c sets using digestion by two acids (hydrochloric and nitric) were excluded as method outliers based on statistical tests, and no sets were received by digestion using three acids

d sets by fusion using lithium metaborate and sodium pyrosulphate were included only, based on statistical tests

e sets by acid and microwave digestion, combustion followed by infrared spectroscopy, fusions, and fused pellet were included

f the data included sets by various fusions and one set by digestion using three acids

g no sets by digestion with either two or three acids were received

Table 2 – WPR-1a Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Cr no AD [a]	%	0.322	0.006	0.025	0.012
In AD [b, c]	µg/g	0.0899	0.0041	0.0041	0.0041
loss on ignition [d]	%	8.42	0.09	0.46	0.26
Mg AD4 + no AD [e]	%	15.22	0.07	0.29	0.18
Na [f]	%	0.050	0.004	0.012	0.006
Nb no AD2 [g]	µg/g	3.88	0.18	0.39	0.30
Ni AD2 + AD3 [h]	%	0.385	0.007	0.051	0.037
Se no AD2 [g]	µg/g	7.7	0.4	1.0	0.8
Sn no AD2 [c, g]	µg/g	1.16	0.17	0.17	0.08
Ta [i]	µg/g	0.242	0.015	0.018	0.015
Te no AD2 [g]	µg/g	0.958	0.058	0.075	0.066
Th no AD2 [g, j]	µg/g	0.64	0.05	0.15	0.09
Tl AD [k]	µg/g	0.0752	0.0048	0.0063	0.0042
Zr FUS [l]	µg/g	41.8	2.1	2.8	2.4

a sets using digestion by any combination of two, three or four acids (hydrochloric, nitric, hydrofluoric and perchloric) were excluded as method outliers based on statistical tests

b sets using digestions by various combinations of acids were included only based on statistical tests

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

d samples of 0.5 to 1 grams ignited for 0.5 to 2 hours at 900 to 1050°C are included only

e sets using digestion by two acids (hydrochloric and nitric) or three acids (hydrochloric, nitric and hydrofluoric) were excluded as method outliers based on statistical tests

f data fulfills the conditions for certified but the element was reclassified as provisional since the between-laboratories standard deviation is about 25% of the mean value

g sets using digestion by two acids (hydrochloric and nitric) were excluded as method outliers based on statistical tests

h sets using digestion by two acids (hydrochloric and nitric) or three acids (hydrochloric, nitric and hydrofluoric) were included only based on statistical tests

i no sets by digestion using two acids were received

j data fulfills the conditions for certified but the element was reclassified as provisional since the between-laboratories standard deviation is 23% of the mean, and 3 sets of data have one significant figure

k sets by various methods using digestion with acids were received only, excluding outliers based on significant figures; data fulfilled the conditions for certification, but the element was re-classified as provisional since all of the accepted data have one significant figure

l sets by fusion with lithium metaborate and sodium pyrosulphate are included only based on statistical tests

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

Analyte	Units	Mean	No. accepted laboratories / values	Analyte	Units	Mean	No. accepted laboratories / values
Al AD2 + AD3 [a]	%	2.3	4 / 20	Mo AD3 + AD4 [f]	µg/g	1.1	6 / 30
Au [b]	µg/g	0.05	17 /85	Mo FUS [g]	µg/g	2	3 / 15
Ba AD2 + AD3 [a]	µg/g	50	5 / 25	moisture [h]	%	0.6	6 / 30
Be AD4 [c]	µg/g	0.2	6 / 30	P AD2 [d]	%	0.03	4 / 20
C	%	0.15	5 / 25	Re [i]	µg/g	0.022	5 / 25
Ca AD2 [d]	%	0.7	4 / 20	Rh [j]	µg/g	0.03	5 / 24
Ce AD2 [d]	µg/g	6	3 / 15	Ru [k]	µg/g	0.06	5 / 24
Co AD2 + AD3 [a]	µg/g	190	7 / 35	Sc AD2 [d]	µg/g	8	3 / 15
Cr AD2 [d]	%	0.08	4 / 20	Se AD2 [d]	µg/g	6	4 / 20
Cr AD4 [c]	%	0.2	4 / 20	Sn AD2 [d]	µg/g	0.7	3 / 15
Fe AD2 [d]	%	9	4 / 20	Sr AD2 + AD3 [a]	µg/g	16	5 / 25
Ga AD2 [d]	µg/g	5	4 / 20	Th AD2 [d]	µg/g	0.3	3 / 15
Ge AD2 [d]	µg/g	0.3	3 / 15	U AD2 [d]	µg/g	0.1	3 / 15
Ge AD4 + FUS [e]	µg/g	1	5 / 25	U AD4 [c]	µg/g	0.2	4 / 20
Hg	µg/g	0.05	3 / 15	U FUS [g]	µg/g	0.3	5 / 25
Ir	µg/g	0.02	7 / 34	V AD2 [d]	µg/g	70	3 / 15
La AD2 + AD3 [a]	µg/g	2.8	4 / 20	W AD4 [c]	µg/g	0.4	3 / 15
Li AD2 + AD3 [a]	µg/g	20	5 / 25	Y AD2 + AD3 [a]	µg/g	5	4 / 16
Mg AD2 + AD3 [a]	%	13	4 / 20	Zn AD2 [d]	µg/g	120	5 / 25
Mn AD2 [d]	%	0.1	4 / 20	Zr AD4 [c]	µg/g	20	5 / 25
Mo AD2 [d]	µg/g	0.9	4 / 20				

a sets using digestion by two acids (hydrochloric and nitric) or three acids (hydrochloric, nitric and hydrofluoric) were included only
b sets with sample weight of 10 grams or less were excluded due to results of assessment of homogeneity
c mean is based on sets by digestion with four acids (hydrochloric, nitric, perchloric and hydrofluoric) only
d sets by digestion using hydrochloric and nitric acids only were included
e sets by digestion using four acids and various fusions only were included
f sets using various acid digestions with three and four acids were included only
g sets using various fusion methods were included only
h samples of 1 to 2 grams dried for 1 or more hours at 105 to 120°C
i sets by digestion only were included
j sets using fire assay pre-concentration only were included
k sets using fire assay pre-concentration with nickel sulphide collection only were included

SOURCE
WPR-1a is a peridotite with rare earth and platinum group elements obtained from the Wellgreen property in the Yukon, Canada. The raw material for WPR-1a was obtained from the same source as its predecessor, WPR-1.

DESCRIPTION
The mineral species include: serpentine (47.4%), talc (11.0%), diopside (9.8%), amphibole (8.7%), pyrrhotite (4.9%), magnetite (4.5%), ferrohornblende (3.5%), phlogopite (2.2%), chlorite (1.6%), pentlandite (1.4%), unnamed mineral: Fe-Cr-Cl (1.2%), calcite (0.9%), ilmenite (0.8%), chalcopyrite (0.7%), other (0.4%), pyrite (0.3%), wilkeite (0.3%), quartz (0.2%), titanite (0.2%), anorthite (0.1%), and albite (0.04%).

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

INSTRUCTIONS FOR USE
WPR-1a should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. 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 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 400-gram units. 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. The gold, platinum and palladium in 10-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, and copper 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.

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 aluminum, cobalt, copper, palladium and platinum. Gold is suspected of having a nugget effect, and thus was not certified.

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, graphite furnace and instrumental neutron activation analysis.

The methods used for 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. Digestion using acids and combustion followed by cold vapour atomic absorption spectrometry and inductively coupled plasma – mass spectrometry was used for the determination of mercury. Sulphur was determined by digestions using acids, microwave digestion, fusions, followed by gravimetric analysis, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry, as well as combustion followed by infrared spectroscopy. Carbon was determined using the latter method exclusively.

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. Forty-five parameters were certified (see Table 1). Many certified elements exclude digestion by two and or three 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

Fourteen 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 indium by acid digestions only and tin by all methods except digestion using two acids. Informational values for 41 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

WPR-1a was released as a new material in April 2012.

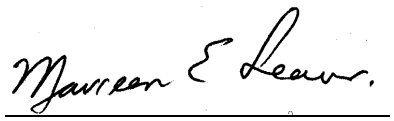
PERIOD OF VALIDITY

The certified values are valid until March 31, 2033. The stability of the material will be monitored every five 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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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.