

CCRMP Canadian Certified Reference Materials Project

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# **Certificate of Analysis**

First issued: January 1994

Version: January 2010

## WPR-1

### Certified Reference Material for an Altered Peridotite with Gold and Platinum Group Elements

Constituent	Unit	Mean	Within-Lab Standard Deviation	Between- Labs Standard Deviation	95% Confidence Limit
Au	ng/g	42.2	6.4	6.2	± 2.8
Cu	%	0.164	0.005	0.013	± 0.008
Fe <sub>2</sub> O <sub>3</sub>	%	14.6	0.4	0.5	± 0.3
lr	ng/g	13.5	1.3	3.2	± 1.8
K <sub>2</sub> O	%	0.12	0.03	0.04	± 0.03
MnO	%	0.166	0.004	0.010	± 0.006
Pd	ng/g	235	21	21	± 9
Pt	ng/g	285	24	29	± 12
Rh	ng/g	13.4	1.6	1.5	± 0.9
Ru	ng/g	21.6	3.3	6.8	± 4.3
TiO <sub>2</sub>	%	0.29	0.02	0.03	± 0.02

Table 1 – Certified Values



Canada

Constituent	Unit	Mean	Within-Lab Standard Deviation	Between- Labs Standard Deviation	95% Confidence Limit
Ag	ug/g	0.7	0.05	0.2	± 0.2
Al <sub>2</sub> O <sub>3</sub>	%	2.95	0.09	0.22	± 0.15
As	ug/g	1.4	0.5	0.9	± 0.8
Ва	ug/g	22	2	7	± 4
Bi	ug/g	0.19	0.05	0.05	± 0.09
CaO	%	2.07	0.07	0.16	± 0.11
Cd	ug/g	0.43	0.16	0.13	± 0.17
Се	ug/g	6	0.4	1	± 1
Со	ug/g	180	5	15	± 9
Cr	%	0.33	0.02	0.09	± 0.05
Cs	ug/g	0.73	0.09	0.06	± 0.06
Dy	ug/g	1.1	0.1	0.3	± 0.3
Er	ug/g	0.5	0.05	0.1	± 0.2
Eu	ug/g	0.31	0.04	0.06	± 0.06
Ga	ug/g	4.5	0.5	0.8	± 0.9
CaO	%	2.07	0.07	0.16	± 0.11
Gd	ug/g	0.9	0.1	0.3	± 0.4
Hf	ug/g	0.61	0.05	0.15	± 0.15
Но	ug/g	0.18	0.03	0.05	± 0.09
La	ug/g	2.2	0.1	0.3	± 0.2
Li	ug/g	4.2	0.2	1.0	± 1.6
Lu	ug/g	0.07	0.008	0.03	± 0.03
MgO	%	31	1	3	± 2
LOI	%	10.2	0.1	0.2	± 0.3
Мо	ug/g	0.9	0.2	0.3	± 0.3
Na₂O	%	0.041	0.008	0.023	± 0.016
Nb	ug/g	2.4	0.2	0.6	± 0.8

Table 2 – Provisional Values

Constituent	Unit	Mean	Within-Lab Standard Deviation	Between- Labs Standard Deviation	95% Confidence Limit
Nd	ug/g	3.5	0.3	0.7	± 0.7
Ni	%	0.29	0.01	0.04	± 0.02
Os	ng/g	13.3	1.6	1.6	± 2.1
P <sub>2</sub> O <sub>5</sub>	%	0.037	0.002	0.011	± 0.012
Pb	ug/g	6	1	3	± 3
Pr	ug/g	0.7	0.05	0.2	± 0.3
Rb	ug/g	5	0.4	2	± 2
S	%	0.94	0.04	0.04	± 0.06
Sb	ug/g	0.9	0.08	0.48	± 0.35
Sc	ug/g	12	0.4	1	± 1
Se	ug/g	4	0.6	1	± 1
SiO <sub>2</sub>	%	36.2	0.9	0.1	± 0.4
Sm	ug/g	0.9	0.06	0.21	± 0.2
Sn	ug/g	1.1	0.2	0.2	± 0.3
Sr	ug/g	7	1.0	2.0	± 2
Th	ug/g	0.4	0.06	0.2	± 0.2
Tm	ug/g	0.09	0.02	0.02	± 0.03
U	ug/g	0.2	0.05	0.10	± 0.1
V	ug/g	65	3	37	± 26
Y	ug/g	5	0.3	1	± 1
Yb	ug/g	0.48	0.05	0.13	± 0.14
Zn	ug/g	95	6	19	± 11
Zr	ug/g	18	3	3	± 3

Table 2 – Provisional Values (cont'd)

Constituent	Unit	Range
В	ug/g	35-130
Be	ug/g	0.1-0.3
CI	ug/g	100-300
Ge	ug/g	1-5
H <sub>2</sub> O <sup>-</sup>	%	0.4-0.6
SO <sub>3</sub>	%	0.5-2.5
Та	ug/g	0.1-0.3
Tb	ug/g	0.1-0.2
Те	ug/g	0.1-0.7
ТІ	ug/g	0.2-0.5
W	ug/g	0.1-3

#### Table 3 - Informational Values

#### DESCRIPTION

The raw material for WPR-1 was obtained from the Wellgreen Complex, Yukon Territory, Canada. WPR-1 was prepared and certified in cooperation with the Geological Survey of Canada (GSC).

WPR-1 is an altered peridotite which contains essentially antigorite with small amounts of chlorite and accessory magnetite and chromite. The peridotite contains pyrrhotite, pentlandite and chalcopyrite all either enclosed, penetrated or intergrown with magnetite. Violarite occurs as inclusions in the pyrrhotite. Tellurides were observed which have been tentatively identified as platinum group element complexes.

The raw material was dried, crushed, grounded, sieved, and blended to obtain a minus 74 micron (200 mesh) product. The yield was 80%. The material comes in glass bottles containing 400g each. This is the only size available.

#### INTENDED USE

WPR-1 is suitable for the analysis of gold, elements from the platinum group and other elements at major, minor and trace levels. Examples of intended use are for quality control in the analysis of samples of a similar type, method development, arbitration and the calibration of equipment.

#### INSTRUCTIONS FOR USE

The assigned values pertain to the date when issued. WPR-1 should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. The material can be stored at room temperature and pressure with no special precautions.

#### 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.

#### LEVEL OF HOMOGENEITY

The homogeneity of the stock with respect to its gold, platinum and palladium was investigated using twenty-two bottles chosen according to the bottling sequence and a stratified random sampling scheme. Two splits were analyzed from each bottle. The analyses for gold, platinum and palladium were

performed by GSC on 10g-samples using fire assay pre-concentration and followed by inductively coupled plasma mass spectrometry.

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 three elements. Use of a smaller mass than indicated will invalidate the use of the certified value and associated parameters.

#### CERTIFIED VALUES

The first interlaboratory measurement program was held in 1992 for the certification of gold and the platinum group elements. Twelve university, government, industrial and commercial laboratories submitted results. In 1994, thirty-three individual laboratories participated in the interlaboratory measurement program in an attempt to certify other elements. Up to 80 elements were analyzed by methods of each laboratory's choice. For gold and the platinum group elements, fire assay, multi-acid digestion followed by solvent extraction, gravimetric, inductively coupled plasma –optical emission spectroscopy, inductively coupled plasma – mass spectroscopy, graphite furnace atomic absorption spectroscopy, direct current plasma spectroscopy, and neutron activation, x-ray fluorescence, hydride generation, inductively coupled plasma – optical emission spectroscopy, direct current plasma spectroscopy, inductively coupled plasma – optical emission spectroscopy, and neutron activation analysis were used. For the other elements, various acid digestions, fusions, gravimetric, combustion, x-ray fluorescence, hydride generation, inductively coupled plasma – optical emission spectroscopy, direct current plasma spectroscopy, and neutron spectroscopy, direct current plasma spectroscopy, and neutron activation analysis were used.

A one-way analysis of variance technique (ANOVA) was used to estimate the consensus value and other statistical parameters (1). The two criteria for certification involve the agreement of within- and between-laboratories standard deviations and the number of sets with acceptable agreement. Table 1 contains the means and associated statistical parameters for the fifteen certified elements. Full details of all phases of the work, including statistical analysis, the methods and the names of the participants are contained in certification report.

#### UNCERTIFIED VALUES

Table 2 contains the provisional elements which did not meet either one or both of the two criteria for certification. Table 3 contains the informational values calculated from the mean of two or more sets of results which were considered to be in good agreement.

#### TRACEABILITY

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

#### DATE OF CERTIFICATION

WPR-1 was released in 1994. The 2004 version of this certificate was written in order to release new or upgraded values. The 2004 version of the certificate included five new certified values, forty-nine new provisional values and eleven new informational values. The 2004 version of the certificate was re-issued in January 2010 with no changes due to the expiration of the former.

#### PERIOD OF VALIDITY

These certified values are valid until January 31, 2032. The stability of the material will be monitored every two years. Updates will be published on the CCRMP web site.

#### LEGAL NOTICE

CANMET - Mining and Mineral Sciences Laboratories (MMSL) 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 - MMSL from and against all liability and costs arising out of the use of this material and information.

**CERTIFYING OFFICER** 

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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.