CCRMP
Canadian Certified Reference Materials Project

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Projet canadien de matériaux de référence certifiés

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Certificate of Analysis

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WMS-1a

Certified Reference Material for Massive Sulphide
with Gold and Platinum Group Elements

Table 1 – WMS-1a Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al	%	1.350	0.021	0.084	0.051
As	µg/g	30.9	2.9	4.8	2.9
Au	µg/g	0.300	0.043	0.040	0.018
Ca*	%	3.09	0.05	0.17	0.11
Cu**	%	1.396	0.014	0.045	0.021
Fe	%	45.4	0.5	1.2	0.6
Ni	%	3.02	0.05	0.15	0.07
Pd	µg/g	1.45	0.05	0.11	0.05
Pt	µg/g	1.91	0.07	0.10	0.05
Rh	µg/g	0.222	0.015	0.052	0.038
S	%	28.17	0.27	0.96	0.69

* Certified value with digestions by two acids excluded as statistical outliers.
** Certified value with digestions by two acids excluded as method outliers based on statistical tests.

Table 2 – WMS-1a Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	3.7	0.2	1.3	0.5
Co	%	0.145	0.002	0.017	0.008
Cr	µg/g	68	3	15	10
K	%	0.0991	0.0034	0.0094	0.0073
Ir*	µg/g	0.322	0.010	0.018	0.019
Mg	%	0.331	0.007	0.035	0.022
Mn	µg/g	600	10	120	70
Na	%	0.0329	0.0034	0.0074	0.0065
Ru*	µg/g	0.145	0.007	0.013	0.015
Sb	µg/g	6.92	1.01	0.98	0.96
Sr	µg/g	31.3	0.7	5.2	4.3
Ti	µg/g	840	20	120	80
V	µg/g	140	6	25	21
Zn	µg/g	130	4	19	8

*Statistical analysis of the results for these elements warrants classification as Provisional, despite only 6 sets for ruthenium and 7 sets for iridium. Ruthenium value is based on nickel sulphide fire assay only.

Table 3 – WMS-1a Informational Values

Analyte	Units	Mean	Number of accepted laboratories / values
Ba	µg/g	70	7 / 35
Bi	µg/g	1.2	3 /15
C	%	0.1	2 / 10
Cd	µg/g	1.4	4 / 20
Ce	µg/g	7.9	4 / 20
Cu (AD2)*	%	1.34	6 / 30
Cs	µg/g	0.6	4 / 20
Dy	µg/g	0.8	3 / 15
Er	µg/g	0.4	3 / 15
Eu	µg/g	0.2	4 / 20
Ga	µg/g	4	3 / 15
Gd	µg/g	0.8	3 /15

Hf	µg/g	0.5	4 / 20
Ho	µg/g	0.2	3 / 15
In	µg/g	0.2	3 / 15
La	µg/g	4.3	5 / 30
Li	µg/g	3	4 / 20
H_2O (105 – 110°C)	%	0.2	2 / 10
LOI **	%	11	2 / 10
Lu	µg/g	0.08	3 / 15
Mo	µg/g	3.0	7 / 35
Nb	µg/g	2.0	3 / 15
Nd	µg/g	4	3 / 15
Os	µg/g	0.15	3 / 12
P	%	0.018	7 / 35
Pb	µg/g	33	18 / 88
Pr	µg/g	1.0	3 / 15
Rb	µg/g	3	3 / 15
Sc	µg/g	3	4 / 25
Se	µg/g	87	7 / 40
Si	%	4.7	6/ 30
Sm	µg/g	0.8	4 / 25
Sn	µg/g	2.3	4 / 20
Ta	µg/g	0.1	3 / 15
Tb	µg/g	0.1	3 / 15
Th	µg/g	1.2	3 / 15
Tm	µg/g	0.08	3 / 15
U	µg/g	0.5	4 / 20
Y	µg/g	4	4 / 20
Yb	µg/g	0.5	4 / 20
Zr	µg/g	20	4 / 20

* Copper by two acid digestion (AD2) only
** Loss on ignition at 1000 – 1050°C

SOURCE

The raw material used to prepare WMS-1a was obtained from the Wellgreen property, near Whitehorse, Yukon. The mine is owned by Northern Platinum Limited. WMS-1a was obtained from the same mine as its predecessor, WMS-1, which is no longer available.

DESCRIPTION

Major species include pyrrhotite (59.7%), clinochlore (11.2%), mainly actinolite plus traces of sepiolite (9.1%), pentlandite (8.8%), clinopyroxene (6.0%), and chalcopyrite (4.1%). Minor species include mica (0.8%), magnetite (0.2%) and galena (0.1%).

INTENDED USE

WMS-1a is suitable for the analysis of gold, platinum group elements and various other elements at major, minor and trace levels in minerals. Examples of intended use include quality control, method development, environmental assessment and the calibration of equipment.

INSTRUCTIONS FOR USE

WMS-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 date when issued. 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, and sieved to remove the plus 74 µm fraction. The product was blended, then bottled in 200-gram units. The yield was 83%. Each bottle was sealed under nitrogen in a laminated aluminum foil-mylar pouch to prevent oxidation.

HOMOGENEITY

The homogeneity of the stock was investigated using twenty-two bottles chosen according to a stratified random sampling scheme. Two splits were analysed from each bottle. Lead fire assay pre-concentration was performed on 10 gram-samples, followed by determination of gold, platinum and palladium by both inductively coupled plasma - optical emission spectrometry and mass spectrometry. Additionally, samples of 0.25–gram were digested with hydrochloric, nitric, perchloric and hydrofluoric acids. Analyses for silver, copper and nickel were performed using inductively coupled plasma - optical emission spectrometry. Inductively coupled plasma - mass spectrometry was used for the determination of lead and zinc. In a third investigation, samples of 0.15-gram were used for the determination of sulphur by combustion.

Use of a smaller sub-sample than specified above will invalidate the use of the certified values and associated parameters. 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 was compared to the F statistic at the 95% level of probability. No evidence of inhomogeneity was observed for these elements.

CERTIFIED VALUES

Thirty-three industrial, commercial, and government laboratories participated in an interlaboratory measurement program using methods of their own choosing. Fire assay, multi-acid digestions, combustion and fusions were used for the concentration step. Inductively coupled plasma – optical emission spectrometry, inductively coupled plasma - mass spectrometry, atomic absorption spectrometry, instrumental neutron activation, x-ray fluorescence, hydride generation, visible and ultraviolet spectrometry and gravimetric analysis were used for the determination step.

ANOVA was used to calculate the consensus values and other statistical parameters[1] 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. Eleven elements 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 document, "Assessment of laboratory proficiency using CCRMP reference materials", at www.ccrmp.ca under Publications, which is based on Guide 33:2000.

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 alternatively**,** more than 8 sets of data that do not fulfill the CCRMP statistical criteria required for certification. Informational values for 41 elements, shown in Table 3, were derived from the means of a minimum of 2 sets.

TRACEABILITY

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

CERTIFICATION HISTORY

WMS-1a is a new material.

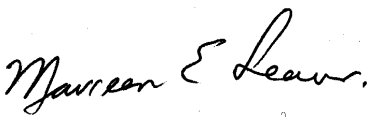
PERIOD OF VALIDITY

The certified values are valid until December 31, 2030. The stability of the material will be monitored every two years for the duration of the inventory. Updates will be made via 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 WMS-1a 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.