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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DS-1

Certified Reference Material for a Gold Ore

Table 1 – DS-1 Certified Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	0.47	0.06	0.12	0.09
Al	%	4.48	0.07	0.24	0.13
As	µg/g	6960	160	300	160
Au	µg/g	32.59	0.56	1.32	0.60
Ba	µg/g	221	7	13	9
Co	µg/g	9.5	0.6	1.2	0.7
Cu	µg/g	27.1	2.0	3.0	1.6
Hg	µg/g	82.0	2.6	5.8	4.0
Mg	%	2.76	0.06	0.13	0.07
Mn	µg/g	437	12	18	10
Ni	µg/g	48.7	1.7	4.0	2.1
P	µg/g	340	16	28	16
Pb	µg/g	13.8	2.3	5.0	3.3
Tl	µg/g	20.0	1.7	1.8	1.5
Zn	µg/g	206	9	18	9

Table 2 – DS-1 Provisional Values

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Be[1]	µg/g	0.819	0.056	0.046	0.052
C[2]	%	3.126	0.015	0.075	0.055
Ca $_{FUSION}$[1]	%	6.248	0.040	0.066	0.063
Ca$_{AD}$[3]	%	5.96	0.13	0.27	0.27
Cd	µg/g	0.98	0.11	0.18	0.14
Cr	µg/g	59	3	12	7
S$_{COMB}$[2]	%	2.609	0.043	0.079	0.063
S$_{AD}$[4]	%	2.85	0.08	0.21	0.18
Sb	µg/g	107	4	16	9
Si	%	25.68	0.20	0.64	0.54
V[5]	µg/g	147.1	3.3	6.6	5.3

1 Statistical analysis of the results warrants classification as Provisional, despite only 7 sets
2 All sets were derived from combustion followed by infrared spectroscopy
3 Statistical analysis of the results warrants classification as Provisional, despite only 7 sets; three and four acid digestions only
4 Two to four acid digestions
5 Digestions by two acids excluded as statistical outliers

Table 3 – DS-1 Informational Values

Element	Units	Mean	Accepted laboratories / values	Element	Units	Mean	Accepted laboratories / values
Bi	µg/g	0.1	3/15	**Lu**	µg/g	0.3	3/15
Ce	µg/g	40	3/15	**Mo**	µg/g	4	4/20
Cs	µg/g	7	4/20	**Na**	µg/g	400	4/20
Eu	µg/g	1	3/15	**Sc**	µg/g	9	5/25
Fe	%	3.0	7/35	**Sm**	µg/g	4	3/15
Ga	µg/g	10	3/15	**Sr**	µg/g	70	4/20
Hf	µg/g	4	3/15	**Ta**	µg/g	0.5	3/15
LOD*	%	0.5	4/17	**Tb**	µg/g	0.5	3/15
In	µg/g	0.05	3/15	**Th**	µg/g	4	3/15
K	%	1.1	5/25	**Ti**	µg/g	2600	5/25
La	µg/g	20	4/20	**U**	µg/g	3	3/15
Li	µg/g	20	4/20	**W**	µg/g	30	5/25
LOI**	%	13	7/32	**Yb**	µg/g	2	3/15

* *loss on drying*
** *loss on ignition*

SOURCE
DS-1 is a gold ore from the Deep Star mine in Carlin Mine Complex, Nevada, USA which was donated by Newmont Mining Corporation.

DESCRIPTION
The mineral species include: quartz (35.5%); feldspars (mainly K-feldspars and lower proportions of albite at 13.6%); dolomite (15.5%); mixed clays (7.5%); mica (mainly muscovite, lower proportions of biotite, trace phlogopite at 6.5%); ankerite (8.5%); pyrite (3.9%); kaolinite (2.8%); calcite (2.4%); pyrrhotite (1.3%); arsenopyrite (0.9%); jarosite (0.5%); garnet (0.3%); gypsum (0.1%); barite (0.2%); apatite, pyrophyllite, realgar, rutile and siderite at 0.1%; and magnetite, sphlerite and wollastonate at trace levels.

INTENDED USE
DS-1 is suitable for the analysis of gold and various other elements at major, minor and trace levels in gold ores. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE
DS-1 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 air dried and dried at 65°C for 48 hours, crushed, milled, sieved to remove the plus 74 µm fraction. The product was blended, and then bottled in 400-gram units. The recovery was 72%.

HOMOGENEITY
The homogeneity of the stock for arsenic, copper, gold and sulphur was investigated using twenty-two bottles chosen according to a stratified random sampling scheme. Two splits were analyzed from each bottle. The splits were analyzed for gold by lead fire assay followed by atomic absorption spectroscopy using a 30–gram sample. Arsenic was determined in 0.1 gram–samples using an aqua regia digest with a microwave followed by determination using inductively coupled plasma – optical emission spectroscopy. Samples of 0.25 grams were used to determine copper using a four-acid digestion and determination by inductively coupled plasma – mass spectrometry. Sulphur was determined on 0.1 gram samples by combustion and infrared detection.

Use of a smaller sub-sample than specified above for these elements 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]. For these four elements the mean squares of the between-bottles variation is similar to or, less than the within-bottle variation. Thus, no significant between-bottle variation was observed for arsenic, copper, gold and sulphur.

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

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. Fifteen elements were certified (see Table 1).

Gold was determined by various fire assay pre-concentration techniques, often with lead, and followed by atomic absorption spectroscopy, inductively coupled plasma - atomic emission spectrometry, or gravimetric finish; or instrumental neutron activation analysis.

Other elements were determined by various multi-acid and microwave digestions, fusions, combustion - infrared spectroscopy, extractions, iron collection followed by atomic absorption spectroscopy, flow injection mass spectrometry, gravimetric analysis, hydride generation atomic absorption spectroscopy, inductively coupled plasma – atomic emission spectroscopy, inductively coupled plasma - mass spectrometry, or titrations. Both fused and pressed powder pellet were used for X-Ray fluorescence spectrometry. Cold vapour separation was used for mercury.

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

Eleven provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CCRMP statistical criterion regarding agreement; or alternatively, 10 or more sets of data, that do not fulfill the CCRMP statistical criteria required for certification. Additionally, the statistical analysis of the data warranted provisional status, despite fewer sets, for beryllium and calcium by fusion and, three and four acid digestions. Informational values for 26 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

DS-1 is a new material.

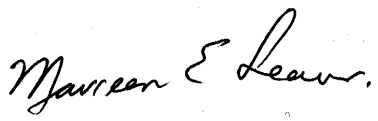
PERIOD OF VALIDITY

The certified values are valid until March 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 DS-1 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.