Certificate of Analysis

First issued: March 2017 Version: March 2017

IOC-1

Certified Reference Material for an Iron Ore

Table 1 – IOC-1 Certified Values

note: Results are based on samples that were dried for one hour at 105°C. The values were generally derived from various fusions and acid digestion methods followed by inductively coupled plasma - optical emission spectroscopy or X-ray fluorescence. Footnotes give details of specific cases. For more detailed information, please refer to the certification report.

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
CaO (non AD) [a, b]	%	0.975	0.007	0.015	0.006
Cr_2O_3	%	0.0224	0.0016	0.0027	0.0013
Fe (T) classical [c]	%	65.66	0.06	0.24	0.16
Fe (T) instr [d]	%	65.62	0.16	0.25	0.13
FeO [c]	%	20.76	0.13	0.29	0.17
Loss on ignition [e]	%	0.863	0.024	0.041	0.020
MgO (non AD) [a]	%	1.043	0.010	0.026	0.011
MnO	%	0.472	0.004	0.012	0.005
P	%	0.0120	0.0006	0.0018	0.0008
SiO_2	%	2.633	0.041	0.048	0.020
TiO_2	%	0.0429	0.0018	0.0052	0.0024

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Table 1 – IOC-1 Certified Values *Cont'd*

a data using various acid digestions was excluded based on statistical tests
b there is some evidence that, under certain conditions, inductively coupled plasma – optical emission spectroscopy gives less precise results
c data was derived from various titrimetric methods
d data was generally derived from various fusions followed by X-ray fluorescence or inductively coupled plasma -optical emission spectroscopy
e samples of 1 to 5 grams were ignited for 1 to 3 hours at 1000 to 1050°C or by thermogravimetry

Table 2 – IOC-1 Provisional values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al_2O_3 [a]	%	0.184	0.009	0.036	0.015
Co [b]	µg/g	19.99	0.51	0.63	0.71
Mo [b]	µg/g	22.89	0.77	0.98	0.96
S [c]	%	0.00657	0.00059	0.00069	0.00045
Sr [b]	µg/g	4.38	0.25	0.30	0.34
V	µg/g	40.8	2.0	8.7	6.3

a data fulfilled the conditions for certified but the element was reclassified as provisional since the between-laboratories standard deviation is approximately 20% of the mean
b statistical analysis of the data warrants classification as provisional despite only 6 sets of data
c data was derived from both combustion - infrared spectrometry and fusion X-ray fluorescence; data fulfilled the conditions for certified but the element was reclassified as provisional since a considerable amount of the data has only one significant figure

Table 3 – IOC-1 Indicative Values (semi-quantitative only)

Analyte	Units	Mean	No. accepted laboratories / values	Analyte	Units	Mean	No. accepted laboratories / values
Ba	µg/g	30	7 / 35	La	µg/g	8	3 / 15
C	%	0.9	4 / 20	MgO (AD) [a]	%	0.9	4 / 18
CaO (AD) [a]	%	0.9	3 / 15	Nb	µg/g	3	4 / 20
Ce	µg/g	14	3 / 15	Ni	µg/g	9	8 / 40

Cont'd

Table 3 – IOC-1 Indicative Values (semi-quantitative only) *Cont'd*

Analyte	Units	Mean	No. accepted laboratories / values	Analyte	Units	Mean	No. accepted laboratories / values
Cl [b]	%	0.003	4 / 20	Pb	µg/g	2	3 / 15
CO_2	%	3	4 / 20	Th	µg/g	0.8	4 / 20
Cu	µg/g	8	6 / 30	W	µg/g	3	3 / 15
Magnetite [c]	%	58	6 / 30	Y	µg/g	3	4 / 20
Ga	µg/g	2	3 / 15	Zn	µg/g	11	7 / 35
K_2O	%	0.004	8 / 38	Zr	µg/g	6	5 / 25

a digestion using various combinations of acids
b data was generally derived from fusion followed by X-ray fluorescence
c data was derived using saturation magnetization analyzer

SOURCE
IOC-1 is an iron ore donated by the Iron Ore Company of Canada, Labrador City, Newfoundland and Labrador, Canada.

DESCRIPTION
The mineral species include: magnetite (58.7%); hematite (31%); siderite (3.1%); dolomite (2.6%); quartz (2.4%); goethite (0.7%); magnesite (0.6%); limonite (0.4%); bixbyite (0.2%); apatite, clays and rhodochrosite (all at 0.1%); garnet and other (both at 0.04%); olivine (0.03%); ankerite, calcite, chamosite, clinochlore and ilmenite (all at 0.02%); albite, biotite, bronzite, eulite, ferrohornblende, hornblende, muscovite and pyrolusite (all at 0.01%).

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

INSTRUCTIONS FOR USE
The contents of the bottle should be thoroughly mixed before taking samples. Samples of IOC-1 should be dried for one hour at 105°C before weighing. 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. CanmetMINING 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 25°C, ground, sieved, mixed and put into bottles each containing 200 grams. The recovery was 57% with a particle size of less than 75 µm (200 mesh). 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 samples were dried for one hour at 105°C. Total iron in samples of 0.25 gram was determined using fusion, separation and titration in a method similar to ISO 7258:1989. Ferrous iron in samples of 0.25 gram was determined using digestion with sulphuric and hydrofluoric acids, the addition of boric acid and titration with potassium dichromate.

Three subsamples for each bottle in a second set of 15 randomly chosen bottles were analyzed. The samples were dried for one hour at 105°C. Samples of 0.5 grams were fused with borate and analyzed by X-ray fluorescence for the determination of calcium, magnesium and silicon.

The evidence indicates that IOC-1 is sufficiently homogeneous for use as a certified reference material. Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters.

CERTIFIED VALUES

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

Methods for the analysis of total iron included ASTM E246-10; ISO 2597-2:2015; ISO 9507: 1990; various fusions or acid digestion followed by various titration methods, inductively coupled plasma – optical emission spectrometry and X-ray fluorescence. Ferrous iron was determined by ASTM E246-10, ISO 9035:1989 and acid digestion followed by titration.

The methods used for various other elements included, in general, various types of fusion or acid digestion follow by flame atomic absorption spectrometry, inductively coupled plasma - optical emission spectrometry, inductively coupled plasma - mass spectrometry and X-ray fluorescence.

Carbon and sulphur were determined by a combustion infrared apparatus, fusion followed by inductively coupled plasma – optical emission spectrometry or ASTM E1019. Fusion followed by X-ray fluorescence was used for the determination of chlorine. Carbon dioxide was determined by combustion infrared apparatus. Loss on ignition was determined using weighing after heating and thermogravimetric analysis.

Magnetite was determined using a saturation magnetic analyzer.

The one-way analysis technique (ANOVA[1]) 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. Eleven means were certified (see Table 1). Two certified elements, calcium and magnesium, exclude acid digestion 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 33:2015, section 8 - 9, and the publication, “Assessment of Laboratory Performance with CCRMP Certified Reference Materials”, at www.ccrmp.ca.

UNCERTIFIED VALUES

Six provisional values (Table 2) were derived from 8 or 9 sets of data that fulfill the CRRMP 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 cobalt, molybdenum and strontium. Informational values for 20 analytes, 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

IOC-1 is a new material.

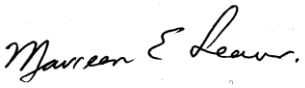
PERIOD OF VALIDITY

The certified values are valid until March 31, 2033. The stability of the material will be monitored every two years for the duration of the inventory.

LEGAL NOTICE

CanmetMINING 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 CanmetMINING 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

In general IOC-1 was prepared in consideration of the principles in ISO Guides 30, 31, 33, 34 and 35. For further details, 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.