Certificate of Analysis

First issued: June 2018   Version: June 2018

REE-3

Certified Reference Material for an Ore with Rare Earth Elements and Zirconium

Table 1 – REE-3 Certified Values

note: Please refer to the footnotes which indicate the analytical methods used to determine certified, provisional or indicative values. For more information, please refer to the certification report.

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Al [a, c]	%	4.372	0.033	0.066	0.034
Ba [a, c]	µg/g	69.1	2.0	4.6	2.1
Be [a]	µg/g	82.3	2.4	5.5	3.2
Ca [a, c]	%	1.644	0.019	0.042	0.019
Ce [c]	µg/g	4,540	80	120	60
Cr [c]	µg/g	82	4	10	5
Cs [a, c]	µg/g	1.118	0.040	0.081	0.048
Cu	µg/g	16.3	1.3	2.8	1.6
Dy [a, c]	µg/g	330.3	5.5	9.5	5.1
Er [a, c]	µg/g	187.2	3.7	4.9	2.7
Eu [a, c]	µg/g	20.85	0.46	0.70	0.36
Fe [a, c]	%	8.28	0.08	0.24	0.10

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Table 1 – REE-3 Certified Values *cont'd*

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Gd [a, c]	**μg/g**	**346**	**8**	**16**	**8**
Hf [a, c]	**μg/g**	**448**	**11**	**16**	**10**
Ho [a, c]	**μg/g**	**65.0**	**1.6**	**1.8**	**0.9**
K [a, c]	**%**	**3.76**	**0.05**	**0.11**	**0.05**
La [c]	**μg/g**	**2,121**	**36**	**45**	**22**
Loss on ignition [b]	**%**	**0.346**	**0.028**	**0.085**	**0.052**
Lu [c]	**μg/g**	**21.53**	**0.47**	**0.66**	**0.37**
Mg [a, c]	**%**	**0.0594**	**0.0029**	**0.0076**	**0.0036**
Mo	**μg/g**	**59.7**	**2.5**	**8.4**	**4.7**
Nb [a, c]	**μg/g**	**1,073**	**30**	**61**	**28**
Nd [c]	**μg/g**	**2,083**	**34**	**52**	**26**
Ni	**μg/g**	**10.83**	**0.90**	**0.90**	**0.58**
P [c]	**%**	**0.0201**	**0.0012**	**0.0036**	**0.0023**
Pb	**μg/g**	**534**	**11**	**24**	**11**
Pr [a, c]	**μg/g**	**550**	**11**	**18**	**9**
Rb [a, c]	**μg/g**	**887**	**14**	**36**	**17**
Si [c]	**%**	**29.66**	**0.11**	**0.11**	**0.04**
Sm [a, c]	**μg/g**	**398**	**8**	**14**	**7**
Sn [a, c]	**μg/g**	**121.1**	**2.7**	**7.5**	**3.6**
Sr [c]	**μg/g**	**133.7**	**3.0**	**8.8**	**3.8**
Ta [a, c]	**μg/g**	**60.7**	**1.7**	**5.6**	**2.7**
Tb [a, c]	**μg/g**	**55.2**	**1.0**	**2.5**	**1.3**
Th [a, c]	**μg/g**	**135.5**	**2.9**	**4.6**	**2.5**
Ti [a, c]	**%**	**0.3202**	**0.0055**	**0.0087**	**0.0038**
Tm [a, c]	**μg/g**	**25.80**	**0.62**	**0.93**	**0.52**
Y [a, c]	**μg/g**	**1,725**	**35**	**96**	**42**

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Table 1 – REE-3 Certified Values *cont'd*

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Yb [a, c]	µg/g	159.4	3.7	4.4	2.4
Zn	µg/g	1,499	29	78	35
Zr [a, c]	µg/g	18,660	160	680	380

a Either no sets were received using digestion by two acids (hydrochloric and nitric acids) or the sets using digestion by two acids were declared method outliers based on statistical tests.

b The mean is based mainly on data from samples of 1 to 2 grams ignited for 1 to 2 hours at 900 to 1050°C.

c The mean is based mainly on data derived from various complete digestion methods.

Table 2 – REE-3 Provisional Values

Analyte	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Bi [a]	µg/g	1.171	0.052	0.085	0.068
Cd	µg/g	4.2	0.2	1.2	0.8
Co [a]	µg/g	0.92	0.07	0.15	0.13
Mn [a, b]	%	0.313	0.004	0.020	0.009
Na [a, b]	%	2.328	0.018	0.076	0.044
Tl	µg/g	2.341	0.073	0.660	0.473
U [a, b]	µg/g	29.9	0.7	3.7	1.8

a Either no sets were received using digestion by two acids (hydrochloric and nitric acids) or the sets using digestion by two acids were declared method outliers based on statistical tests.

b The mean is based mainly on data derived from various complete digestion methods.

Table 3 – REE-3 Indicative Values (semi-quantitative only)

Analyte	Units	Mean	No. accepted laboratories / values
Ag	µg/g	2	8 / 40
C [a]	%	0.08	7 / 35
Ga [e]	µg/g	80	18 / 90
In [b]	µg/g	0.4	5 / 25
Li [b]	µg/g	60	9 / 45
Moisture [c]	%	0.1	6 / 30
S [d]	%	0.04	14 / 70
Sb	µg/g	0.2	5 / 25
Sc	µg/g	3	8 / 40
V	µg/g	4	8 / 40
W [b, e]	µg/g	1	8 / 40

a The mean is based on data derived from combustion followed by infrared spectroscopy only.
b Either no sets were received using digestion by two acids (hydrochloric and nitric acids) or the sets using digestion by two acids were declared method outliers based on statistical tests.
c The data was obtained from samples of 1 to 4 grams dried for 0.7 to 3 hours at 100 - 105°C.
d The mean is based on data derived from digestion using four acids (nitric, hydrochloric, hydrofluoric and perchloric acids) or combustion, followed by various determinations methods.
e The mean is based mainly on data derived from various complete digestion methods.

SOURCE

REE-3 is an ore with rare earth elements and zirconium donated by a Canadian mining company.

DESCRIPTION

The mineral species include: quartz (25.8%); orthoclase (21.0%); albite (15.2%); aegerina-augita (9.8%); aegirine (7.6%); hematite/magnetite (5.0%); zircon (4.3%); biotite (4.2%); allanite-(La) (2.6%); gadolinita-(Sm) (1.7%); murataita and fluorite (0.5%); calcite (0.4%); titanite (0.3%); columbite (0.2%); lourenswalsita, illmenite, chromferide, sphalerite, anorthite and granate (all at 0.1%); fergusonite (0.07%); bastnasite and ekanite (each at 0.06%); apatite and allanite-(Ce) (each at 0.04%); ferrosaponite and pyrite (each at 0.02%); and actinolite, ferro-actinolite, wollastonite, parisite and monazite (each at 0.01%).

INTENDED USE

REE-3 is suitable for the analysis of rare earth elements and other elements in ores at concentrations ranging from major, minor to trace levels. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE

REE-3 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. 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 crushed, ground and sieved. The recovery of the minus 75 µm (200 mesh) fraction was 58%. The product was blended and bottled in 100-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. Subsamples of 0.1 grams were fused with a mixture of lithium metaborate and lithium tetraborate, and analyzed by inductively coupled plasma – atomic emission spectroscopy for calcium, lanthanum and zirconium; and by inductively coupled plasma – mass spectrometry for cerium, niobium and thulium.

A one–way analysis of variance technique (ANOVA)[1] and statistical analyses were used to assess the homogeneity of these elements. No significant between-bottles variation was observed for all elements.

Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters.

CERTIFIED VALUES

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

Methods for the determination of the elements included preparation with various combinations of acids or various types of fusions followed by flame atomic absorption spectroscopy, inductively coupled plasma – atomic emission spectroscopy and inductively coupled plasma – mass spectrometry. X-ray fluorescence on a pressed powder pellet, fused pellet or after a fusion was performed for the determination of many elements.

The concentration of carbon was determined using combustion followed by infrared spectrometry.

The concentration of sulphur was determined using combustion followed by infrared spectrometry; and digestion with various combinations of acids or fusion with sodium peroxide followed by inductively coupled plasma – atomic emission spectroscopy.

ANOVA was used to calculate the consensus values and other statistical parameters from the data 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-one analytes 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 33:2015, sections 8-9, and the publication, “Assessment of laboratory proficiency using CCRMP reference materials”

UNCERTIFIED VALUES

Seven 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. Indicative values for 11 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

REE-3 was released as a new material in June 2018.

PERIOD OF VALIDITY

The certified values are valid until June 30, 2038.

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

Maureen E. Leaver – CCRMP Coordinator

Lyne Lortie – Data Processor

FOR FURTHER INFORMATION

In general REE-3 was prepared in consideration of the principles in ISO Guides 30, 31, 33, 34 and 35. The Certification Report is available free of charge upon request to:

REFERENCES

1. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960.