



**CCRMP**  
Canadian Certified Reference Materials Project



**PCMRC**  
Projet canadien de matériaux de référence certifiés

# Certificate of Analysis

First issued: April 2014

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## REE-1

**Certified Reference Material for Rare Earth Elements,  
Zirconium and Niobium**

**Table 1 – REE-1 Certified Values**

Element	Units	Mean	Within-lab Standard Deviation	Between- labs Standard Deviation	95% Confidence Interval of Mean
Al <sup>a</sup>	%	3.59	0.04	0.12	0.06
As <sup>a</sup>	µg/g	124	5	20	12
Ba <sup>a</sup>	µg/g	100.1	4.5	9.4	4.3
Ca <sup>a</sup>	%	2.30	0.03	0.10	0.05
Ce <sup>b</sup>	µg/g	3960	70	150	70
Co <sup>a</sup>	µg/g	1.58	0.15	0.21	0.13
Cr no AD <sup>b</sup>	µg/g	277	9	35	19
Cs <sup>a</sup>	µg/g	1.07	0.05	0.14	0.08
Cu	µg/g	79.7	2.5	7.6	4.4
Dy <sup>a</sup>	µg/g	847	16	41	19
Er	µg/g	701	13	58	26
Eu <sup>a</sup>	µg/g	23.5	0.6	1.7	0.8
Gd <sup>a</sup>	µg/g	433	10	21	10
Hf <sup>a</sup>	µg/g	479	9	26	14
Ho	µg/g	208	4	21	9
K <sup>a</sup>	%	3.09	0.04	0.13	0.06
La <sup>a</sup>	µg/g	1661	28	72	32
Na <sup>a</sup>	%	1.445	0.021	0.077	0.034
Nb <sup>a</sup>	µg/g	4050	90	350	150
Nd <sup>a</sup>	µg/g	1456	25	53	25
Ni	µg/g	24.7	1.7	3.2	2.0

*cont'd*

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

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
P <sup>a</sup>	%	0.0261	0.0024	0.0053	0.0033
Pb	µg/g	1137	26	99	46
Pr <sup>a</sup>	µg/g	435	8	18	8
Rb <sup>a</sup>	µg/g	1047	24	47	26
Si <sup>a</sup>	%	31.36	0.16	0.38	0.22
Sm <sup>a</sup>	µg/g	381	8	18	8
Sn <sup>a</sup>	µg/g	498	12	52	25
Sr <sup>a</sup>	µg/g	129	3	13	6
Tb <sup>a</sup>	µg/g	106.2	2.0	9.6	4.4
Th <sup>a</sup>	µg/g	719	19	56	26
Tm <sup>a</sup>	µg/g	106.0	2.1	5.8	2.9
U <sup>a</sup>	µg/g	137	3	10	5
Y <sup>b</sup>	µg/g	5480	100	280	130
Yb <sup>a</sup>	µg/g	678	11	50	22
Zr no AD <sup>b</sup>	%	1.91	0.03	0.15	0.07

*a either no sets were received using digestion by two acids (hydrochloric and nitric) and/or by three acids (hydrochloric, nitric and hydrofluoric) or the set(s) by either of these methods was/were declared method outlier(s) based on statistical tests*

*b the data includes sets by digestion using four acids (hydrochloric, nitric, hydrofluoric and perchloric) in a closed vessel, various types of fusions, pressed powder pellet and/or instrumental neutron activation analysis based on statistical tests or the selection of methods by the laboratories*

**Table 2 – REE-1 Provisional Values**

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Be <sup>a</sup>	µg/g	590	9	61	34
Bi <sup>b</sup>	µg/g	0.652	0.042	0.062	0.047
C <sup>c</sup>	%	0.0786	0.0046	0.0095	0.0074
Fe <sup>a</sup>	%	4.16	0.03	0.15	0.06
Ga <sup>a</sup>	µg/g	64	1	11	6
Li <sup>a</sup>	µg/g	205	5	17	12

*Cont'd*

**Table 2 – REE-1 Provisional Values** *cont'd*

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Lu <sup>a</sup>	µg/g	92.4	1.4	8.1	3.8
Mg <sup>a</sup>	%	0.895	0.014	0.063	0.027
Mn <sup>a</sup>	%	0.155	0.003	0.021	0.009
Mo	µg/g	36.6	0.9	4.4	2.3
Sb <sup>a</sup>	µg/g	3.16	0.16	0.17	0.15
Ta <sup>d</sup>	µg/g	231	6	29	13
Ti <sup>a</sup>	%	0.384	0.007	0.030	0.013
Tl <sup>a</sup>	µg/g	1.85	0.07	0.34	0.25
V <sup>a,e</sup>	µg/g	9.9	1.8	4.0	2.6
Zn <sup>a</sup>	µg/g	1870	40	200	90

- a either no sets were received using digestion by two acids (hydrochloric and nitric) and/or by three acids (hydrochloric, nitric and hydrofluoric) or the set(s) by either of these methods was/were declared method outlier(s) based on statistical tests*
- b 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*
- c all of the data was obtained from a combustion apparatus with detection by infrared spectroscopy*
- d the data includes sets by digestion using four acids (hydrochloric, nitric, hydrofluoric and perchloric) in a closed vessel, various types of fusions, pressed powder pellet and/or instrumental neutron activation analysis based on statistical tests or the selection of methods by the laboratories*
- e data fulfills the conditions for certified but the element was reclassified as provisional since the between-laboratories standard deviation is 40% of the mean*

**Table 3 – REE-1 Informational Values (semi-quantitative only)**

Analyte	Units	Mean	No. accepted laboratories / values
Cr (AD3+AD4) <sup>a</sup>	µg/g	230	5 / 25
Ge <sup>b</sup>	µg/g	3	5 / 25
In <sup>b</sup>	µg/g	0.2	4 / 20
loss on ignition <sup>c</sup>	%	2	14 / 70
moisture <sup>d</sup>	%	0.6	10 / 50
S	%	0.03	6 / 30
Sc <sup>e</sup>	µg/g	8	3 / 15
W <sup>b</sup>	µg/g	10	13 / 65

*Cont'd*

### **Table 3 – REE-1 Informational Values (semi-quantitative only) *cont'd***

- a** *data was derived from digestions by three acids (hydrochloric, nitric and hydrofluoric) and four acids (hydrochloric, nitric, hydrofluoric and perchloric)*
- b** *either no sets were received using digestion by two acids (hydrochloric and nitric) and/or by three acids (hydrochloric, nitric and hydrofluoric) or the set(s) by either of these methods was/were declared method outlier(s) based on statistical tests*
- c** *data is based on samples of 1 to 5 grams ignited for 0.8 to 8 hours at 900 to 1100°C; data fulfills the conditions for provisional but was reclassified as informational due to a bi-modal distribution*
- d** *data is based on (i) samples of 0.25 to 5 grams dried for 1 to 22 hours at 90 to 105°C, and (ii) combustion infrared spectroscopy; data fulfills the conditions for provisional but the parameter was reclassified as informational due to the between-laboratory standard deviation being 23% of the mean*
- e** *most sets indicated a limit of detection only; three laboratories gave data by four acid digestion followed by inductively coupled plasma - mass spectroscopy*

#### **SOURCE**

REE-1 is an ore containing rare earth elements (REEs), zirconium and niobium from the Strange Lake deposit, Quebec, Canada. The raw material was donated by Quest Rare Minerals Limited.

#### **DESCRIPTION**

The mineral species include: quartz (39.5%), K-feldspar (22.9%), amphiboles plus pyroxenes (9.3%), albite (5.7%), chlorite (3.1%), gittinsite (2.2%), zircon (1.8%), fluorite and titanite (each at 1.4%), epidote, gittinsite-Y and talc (each at 1.2%), gadolinite-Y and zircon-Y (each at 1.1%), kainosite-Y and pyrochlore-Y (each at 1.0%), anorthite (0.8%), geronite-Y, hematite and UN1930-01 (an oxide) (each at 0.6%), bastnaesite-Y (0.5%), other various unspecified minerals (0.4%), calcite and thorite-like (each at 0.3%), apatite and monazite (each at 0.2%), latrapite, pyrochlore and sphalerite (each at 0.1%), pyrite (0.04%), siderite (0.03%) and ankerite (0.02%).

#### **INTENDED USE**

REE-1 is suitable for the analysis of rare earth elements, zirconium, niobium and various other elements in ores in concentrations ranging from major to minor to trace levels. Examples of intended use include quality control and method development.

#### **INSTRUCTIONS FOR USE**

REE-1 should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. 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 ground and sieved to remove the plus 75 µm fraction. The recovery of the minus 75 µm fraction was 62%. The product was blended, and then 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. For aluminum, calcium, erbium, holmium, silicon and yttrium, test portions of 0.1 grams were fused with lithium metaborate and the concentration was determined using inductively coupled plasma – optical emission spectroscopy or

inductively coupled plasma – mass spectrometry. For niobium and zirconium, test portions of 0.5 grams were fused with lithium tetraborate and analyzed by X-ray fluorescence. Use of a smaller subsample than specified above will invalidate the use of the certified values and associated parameters.

Statistical analyses were used to assess the homogeneity of these elements. No significant between-bottle variation was observed for any of these elements.

### **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 digestion with various combinations of acids and various types of fusions followed by the determination using atomic absorption spectrometry, inductively coupled plasma – optical emission spectroscopy and inductively coupled plasma – mass spectrometry. X-ray fluorescence on a pressed pellet was also used for many elements. Instrumental neutron activation analysis was also used.

The concentrations of carbon and sulphur were determined using a combustion apparatus followed by infrared spectrometry. Additionally, the concentration of sulphur was determined using various fusions followed by X-ray fluorescence.

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's statistical criterion regarding the agreement of the results. Thirty-six (36) elements were certified (see Table 1). Some certified elements exclude some or all types of digestions using acids 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 33:2000, pages 14-17, and the publication, "Assessment of laboratory proficiency using CCRMP reference materials", at [www.ccrmp.ca](http://www.ccrmp.ca).

### **UNCERTIFIED VALUES**

Sixteen (16) 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. Informational values for eight (8) parameters, 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-1 was released as a new material in April 2014.

### **PERIOD OF VALIDITY**

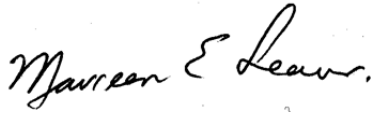
The certified values are valid until April 30, 2044.

### **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



Joseph Salley – Data Processor

**FOR FURTHER INFORMATION**

The Certification Report is available free of charge upon request to:

**CCRMP**  
**CanmetMINING (NRCan)**  
**555 Booth Street, room 433**  
**Ottawa, Ontario, Canada K1A 0G1**  
**Telephone: (613) 995-4738**  
**Facsimile: (613) 943-0573**  
**E-mail: [ccrmp@nrcan.gc.ca](mailto:ccrmp@nrcan.gc.ca)**