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CCRMP
Canadian Certified Reference Materials Project

PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: March 2022 Version: March 2022

SY-5

Certified Reference Material for a Syenite

Table 1 - SY-5 Certified Values

note: The certified, provisional and indicative values herein pertain to the material on an as-received basis. Values for the elements were generally derived from a variety of digestions with mixtures of acids and/or fusions, followed by instrumental analysis. The footnotes indicate the details of the analytical methods used to determine the values. Values for most elements by acid digestion do not include digestion with a mixture of hydrochloric and nitric acids. For more detailed information, please refer to the certification report.

Analyte	Codes for Predominant Methods (see footnotes)	Units	Mean	Within-laboratory Standard Deviation	Between-laboratories Standard Deviation	95% Confidence Interval of Mean
Al_2O_3 [a]	FUS ICP	%	14.35	0.22	0.31	0.22
Al_2O_3 [b]	FB XRF	%	14.18	0.06	0.13	0.07
Ba [b, c]	FB XRF, AD3/4 FUS ICP	µg/g	6480	120	260	90
Be [c]	AD3/4 FUS ICP	µg/g	4.62	0.19	0.34	0.20
C [d]	COMB IR	%	0.171	0.005	0.014	0.008
CaO [e]	AD3/4 ICP	%	6.88	0.07	0.17	0.12
CaO [a]	FUS ICP	%	7.12	0.11	0.22	0.15
CaO [b]	FB XRF	%	7.039	0.027	0.034	0.022
Ce [c]	AD3/4 FUS ICP	µg/g	459	11	27	12

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Table 1 - SY-5 Certified Values *cont'd*

Analyte	Codes for Predominant Methods (see footnotes)	Units	Mean	Within-laboratory Standard Deviation	Between-laboratories Standard Deviation	95% Confidence Interval of Mean
Co [f]	AD2/3/4 FUS ICP	µg/g	23.1	0.7	1.9	0.8
Cr [a, b]	FUS ICP, FB XRF	µg/g	151.5	7.4	7.4	3.3
Cs [c]	AD3/4 FUS ICP	µg/g	0.612	0.034	0.046	0.024
Cu [f]	AD2/3/4 FUS ICP	µg/g	37.9	1.3	2.2	0.9
Dy [c]	AD3/4 FUS ICP	µg/g	11.33	0.28	0.39	0.20
Er [c]	AD3/4 FUS ICP	µg/g	5.38	0.18	0.27	0.13
Eu [c]	AD3/4 FUS ICP	µg/g	7.64	0.20	0.48	0.24
Fe_2O_3 [a]	FUS ICP	%	10.61	0.16	0.16	0.11
Fe_2O_3 [b]	FB XRF	%	10.481	0.057	0.083	0.048
Ga [c]	AD3/4 FUS ICP	µg/g	21.6	0.6	2.0	0.9
Gd [c]	AD3/4 FUS ICP	µg/g	19.8	0.5	1.4	0.7
Hf [a]	FUS ICP	µg/g	15.3	0.8	2.0	1.4
Ho [c]	AD3/4 FUS ICP	µg/g	2.04	0.07	0.13	0.06
K_2O [b]	FB XRF	%	4.150	0.020	0.040	0.023
K_2O [c]	AD3/4 FUS ICP	%	4.170	0.058	0.110	0.052
La [c]	AD3/4 FUS ICP	µg/g	225	5	11	5
Li [c]	AD3/4 FUS ICP	µg/g	49.5	0.8	2.9	1.6
Loss on ignition [g]	GRV1	%	0.86	0.03	0.13	0.06
Lu [c]	AD3/4 FUS ICP	µg/g	0.702	0.026	0.051	0.027
MgO [b]	FB XRF	%	3.305	0.019	0.044	0.025
MgO [e]	AD3/4 ICP	%	3.13	0.04	0.11	0.08
MgO [a]	FUS ICP	%	3.235	0.050	0.085	0.056
MnO [b, c]	FB XRF, AD3/4 FUS ICP	%	0.1283	0.0029	0.0064	0.0022
Mo [f]	AD2/3/4 FUS ICP	µg/g	8.93	0.30	0.69	0.31
Moisture [i]	GRV2	%	0.142	0.019	0.070	0.048

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Table 1 - SY-5 Certified Values *cont'd*

Analyte	Codes for Predominant Methods (see footnotes)	Units	Mean	Within-laboratory Standard Deviation	Between-laboratories Standard Deviation	95% Confidence Interval of Mean
Na_2O [c]	AD3/4 FUS ICP	%	4.15	0.05	0.12	0.06
Nb [c]	AD3/4 FUS ICP	µg/g	22.2	0.8	1.6	0.7
Nd [c]	AD3/4 FUS ICP	µg/g	208.1	4.7	6.9	3.5
Ni [b, c]	FB XRF, AD3/4 FUS ICP	µg/g	79.8	3.0	6.1	2.5
P_2O_5 [c]	AD3/4 FUS ICP	%	2.04	0.03	0.12	0.06
Pb [b, c]	FB XRF, AD3/4 FUS ICP	µg/g	65.9	2.2	5.6	2.5
Pr [c]	AD3/4 FUS ICP	µg/g	55.3	1.2	2.5	1.3
Rb [c]	AD3/4 FUS ICP	µg/g	76.9	2.1	5.8	2.5
S [c, d]	AD3/4 FUS ICP, COMB IR	%	0.421	0.011	0.017	0.008
Sb [f]	AD2/3/4 FUS ICP	µg/g	0.524	0.080	0.080	0.054
Sc [c]	AD3/4 FUS ICP	µg/g	12.37	0.41	0.71	0.36
SiO_2 [b, h]	FB XRF, FUS GRV	%	49.83	0.16	0.36	0.20
SiO_2 [a]	FUS ICP	%	50.1	0.6	1.5	1.1
Sm [c]	AD3/4 FUS ICP	µg/g	31.1	0.8	1.3	0.6
Sn [c]	AD3/4 FUS ICP	µg/g	2.56	0.13	0.19	0.11
Sr [b, c]	FB XRF, AD3/4 FUS ICP	µg/g	3100	40	130	50
Ta [c]	AD3/4 FUS ICP	µg/g	1.06	0.08	0.16	0.08
Tb [c]	AD3/4 FUS ICP	µg/g	2.26	0.07	0.17	0.09
Th [c]	AD3/4 FUS ICP	µg/g	25.2	0.8	1.3	0.5
TiO_2 [c]	AD3/4 FUS ICP	%	1.777	0.028	0.058	0.027
TiO_2 [b]	FB XRF	%	1.810	0.015	0.039	0.022
Tl [c]	AD3/4 FUS ICP	µg/g	0.614	0.030	0.070	0.040
Tm [c]	AD3/4 FUS ICP	µg/g	0.725	0.030	0.040	0.022
U [c]	AD3/4 FUS ICP	µg/g	5.09	0.16	0.38	0.17

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Table 1 - SY-5 Certified Values *cont'd*

Analyte	Codes for Predominant Methods (see footnotes)	Units	Mean	Within-laboratory Standard Deviation	Between-laboratories Standard Deviation	95% Confidence Interval of Mean
V [b, c]	FB XRF, AD3/4 FUS ICP	µg/g	131.8	3.1	9.8	4.0
Y [c]	AD3/4 FUS ICP	µg/g	56.3	1.4	2.9	1.2
Yb [c]	AD3/4 FUS ICP	µg/g	4.64	0.13	0.17	0.09
Zn [b, c]	FB XRF, AD3/4 FUS ICP	µg/g	130.9	3.7	9.1	3.9
Zr [a, b]	FUS ICP, FB XRF	µg/g	769	30	93	45

a FUS ICP: the mean is based mainly on data derived from fusion with various fluxes followed by inductively coupled plasma - optical emission spectroscopy or mass spectrometry.

b FB XRF: the mean is based mainly on data derived from fused bead prepared with various fluxes followed by X-ray fluorescence spectroscopy.

c AD3/4 FUS ICP: the mean is based mainly on data derived from both various digestions with mixtures of three or four acids or fusion with various fluxes followed by inductively coupled plasma - optical emission spectroscopy or mass spectrometry. Either no sets were received using digestion by two acids (hydrochloric and nitric) or the set(s) was/were declared method outlier(s) based on statistical tests.

d COMB IR: the mean is based on data derived from combustion apparatus with infrared spectroscopy.

e AD3/4 ICP: the mean is based mainly on data derived from various digestions with mixtures of three or four acids followed by inductively coupled plasma - optical emission spectroscopy or mass spectrometry. Either no sets were received using digestion by two acids (hydrochloric and nitric) or the set(s) was/were declared method outlier(s) based on statistical tests.

f AD2/3/4 FUS ICP: the mean is based mainly on data derived from both various digestions with mixtures of two, three or four acids or fusion with various fluxes followed by inductively coupled plasma - optical emission spectroscopy or mass spectrometry.

g GRV1: the mean is based on data from samples of 1 to 2.5 grams ignited for 1 to 16 hours at 1000 to 1050°C.

h FUS GRV: the mean is based on data derived from fusion with various fluxes followed by a gravimetric determination.

i GRV2: the mean is based on data from samples of 1 to 3 grams dried for 1 - 24 hours at 100 – 105°C.

Table 2 - SY-5 Provisional Values

Analyte	Codes for Predominant Methods (see footnotes)	Units	Mean	Within-laboratory Standard Deviation	Between-laboratories Standard Deviation	95% Confidence Interval of Mean
Al_2O_3 [a]	AD3/4 ICP	%	13.96	0.14	0.32	0.25
Bi [b]	AD2/3/4 ICP	µg/g	0.083	0.011	0.011	0.009
Cr [a]	AD3/4 ICP	µg/g	130.7	4.8	6.0	4.9
Fe_2O_3 [a]	AD3/4 ICP	%	10.19	0.12	0.41	0.32
Hf [a, c]	AD3/4 ICP	µg/g	5.11	0.18	0.26	0.25
In [b]	AD2/3/4 ICP	µg/g	0.0856	0.0065	0.0065	0.0052
Na_2O [d]	FB XRF	%	4.14	0.02	0.11	0.06
P_2O_5 [d]	FB XRF	%	2.012	0.010	0.047	0.026

a AD3/4 ICP: the mean is based mainly on data derived from various digestions with mixtures of three or four acids followed by inductively coupled plasma - optical emission spectroscopy or mass spectrometry. Either no sets were received using digestion by two acids (hydrochloric and nitric) or the set(s) was/were declared method outlier(s) based on statistical tests.

b AD2/3/4 ICP: the mean is based mainly on data derived from both various digestions with mixtures of two, three or four acids followed by inductively coupled plasma – optical emission spectroscopy or mass spectrometry.

c Statistical analysis of the data warrants classification as provisional despite only 7 sets of data.

d FB XRF: the mean is based mainly on data derived from fused bead prepared with various fluxes.

Table 3 - SY-5 Indicative Values (semi-quantitative only)

Analyte	Codes for Predominant Methods (see footnotes)	Units	Mean	No. Accepted Laboratories / Values
Ag [a]	AD2/3/4 ICP	µg/g	0.3	11 / 55
As [a]	AD2/3/4/ ICP	µg/g	1	8 / 40
Cd [a]	AD2/3/4 ICP	µg/g	0.08	9 / 45
Ge [b]	AD2/3/4 FUS ICP	µg/g	1	10 / 50
Te [a]	AD2/3/4 ICP	µg/g	0.03	4 / 20
Zr [c]	AD3/4 ICP	µg/g	230	8 / 40

a ***AD2/3/4 ICP: the mean is based mainly on data derived from various digestions with mixtures of two, three or four acids followed by inductively coupled plasma – optical emission spectroscopy or mass spectrometry.***

b ***AD2/3/4 FUS ICP: the mean is based mainly on data derived from both various digestions with mixtures of two, three or four acids, or fusion with various fluxes followed by inductively coupled plasma - optical emission spectroscopy or mass spectrometry.***

c ***AD3/4 ICP: the mean is based mainly on data derived from various digestions with mixtures of three or four acids followed by inductively coupled plasma – optical emission spectroscopy or mass spectrometry. Either no sets were received using digestion by two acids (hydrochloric and nitric) or the set(s) was/were declared method outlier(s) based on statistical tests.***

SOURCE

SY-5 is a rock obtained from an outcrop near the Township of Admaston/Bromley, Ontario, Canada.

DESCRIPTION

The mineral species include: albite (25.1%), kaersutite (19.2%), microcline (18.8%), hematite/magnetite (8.5%), apatite (5.4%), titanite (5.2%), biotite (3.7%), pyrite (1.8%), magnesiogedrite (1.0%), actinolite (0.97%), calcite (0.6%), anorthite (0.6%), quartz (0.6%), zircon (0.4%), garnet-pyrope (0.3%), barite (0.1%), plagioclase (0.1%), allanite-(Ce) (0.1%), ilmenite (0.1%), and unclassified (7.5%).

INTENDED USE

SY-5 is suitable for the analysis of elements in rocks at major, minor and trace levels of concentration. Examples of intended use include quality control and method development.

INSTRUCTIONS FOR USE

SY-5 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, milled and sieved. The recovery of the fraction less than 75 µm (minus 200 mesh) was 67%. 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. The major elements selected were, calcium, iron, silicon and titanium. Subsamples of 1 gram were fused with a mixture of lithium tetraborate and lithium metaborate, and analyzed by X-ray fluorescence spectroscopy. Three additional subsamples of 0.1 gram from each bottle were analyzed for chromium, strontium, yttrium and zinc by fusion with lithium metaborate, lithium tetraborate and lithium bromide followed by determination by inductively coupled plasma optical emission spectroscopy.

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

Methods for the determination of the concentration of the elements included mainly preparation with various combinations of acids or various types of fusions followed by the determination with inductively coupled plasma optical emission spectroscopy and inductively coupled plasma mass spectrometry. Additionally, the concentration of some elements was determined by the preparation of a fused bead prepared with various fluxes followed by X-ray fluorescence.

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

The concentration of sulphur was determined mainly using (i) combustion followed by infrared spectrometry, and (ii) digestion with various combinations of acids or fusion followed by inductively coupled plasma optical 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. Sixty-three 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", at www.ccrmp.ca.

UNCERTIFIED VALUES

Eight 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; or 7 sets of data for which the statistical analysis of the data warranted provisional status. Indicative values for six analytes, shown in Table 3, were derived from the means of a minimum of 4 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 complementary report for this material provides the available details.

CERTIFICATION HISTORY

SY-5 was released as a new material in March 2022.

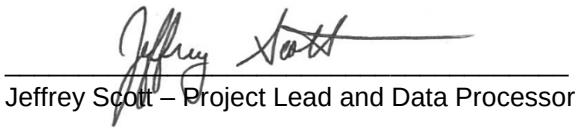
PERIOD OF VALIDITY

The certified values are valid until March 31, 2042.

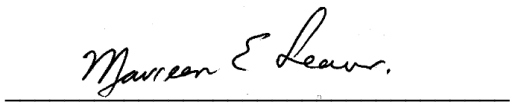
LEGAL NOTICE

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



Jeffrey Scott – Project Lead and Data Processor



Maureen E. Leaver – CCRMP Coordinator

FOR FURTHER INFORMATION

SY-5 was prepared in consideration of the principles in ISO 17034:2016 and ISO Guides 30, 31, 33 and 35. 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.