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CERTIFICATE OF ANALYSIS

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ACCREDITED
CERTIFICATE #4015.01

RMMG-2

Mining Waste (Tertiary mud)

Original certificate: December, 2016
Revision: December, 2016

RMMG-2 is a tertiary mud sample, the residue generated by the Votorantim Metais primary zinc processing unit, located in Três Marias, Minas Gerais, Brazil. The raw material was oven-dried and pulverized to pass a 0,075 mm screen and then homogenized. This reference material is intended for use in calibration of a measurement system, assessment of a measurement procedure, quality control and value assignment to materials with similar matrices. An RMMG-2 unit consists of around 90 g of material packaged in eight vacuum sealed PET+aluminum+PE lined sachets, containing a minimum of 11 g of material each.

This material was characterized by means of an interlaboratory program involving seventeen competent laboratories using a variety of methods of demonstrable accuracy. Robust statistical methods [1] were used to estimate the property values and variability components. Certified values were assigned based on a minimum of five accepted data sets, analytical measurement methods suitable for the property value and fitness of the uncertainty associated with the property value for the intended use. The stated uncertainty is an expanded uncertainty, with coverage factor 2, estimated by the uncertainty due to material characterization.

Certified Values

Constituent	Unit	Mass fraction	Repeatability standard deviation	Reproducibility standard deviation	No. of data sets	Minimum sample (g)[*1]	Analytical methods
Ag	mg/kg	75,1 ± 2,9	2,9E+00	5,4E+00	21	0,1	a, b, c, d, j
Al	%(m/m)	2,913 ± 0,044	6,4E-02	8,5E-02	23	0,1	b, c, e, i
As	mg/kg	162,1 ± 5,4	7,0E+00	1,0E+01	23	0,1	b, c, d, j
Ba	mg/kg	429 ± 15	1,6E+01	2,4E+01	16	0,1	c, d, e, i, j
Be	mg/kg	2,19 ± 0,25	2,1E-01	3,0E-01	9	0,1	b, c, g
Bi	mg/kg	15,24 ± 0,50	7,2E-01	8,5E-01	18	0,1	b, c, d, f
Ca	%(m/m)	7,15 ± 0,12	1,6E-01	2,7E-01	33	0,1	b, c, e, h, i, j
Cd	mg/kg	234,9 ± 8,0	7,4E+00	1,4E+01	20	0,1	b, c, d
C	%(m/m)	0,287 ± 0,014	1,4E-02	1,8E-02	11	0,1	k
Ce	mg/kg	34,1 ± 1,0	2,3E+00	1,6E+00	15	0,1	b, c, d, j
Co	mg/kg	29,56 ± 0,97	1,6E+00	1,9E+00	24	0,1	b, c, d, i, j
Cr	mg/kg	47,3 ± 3,4	2,5E+00	4,5E+00	11	0,1	b, c, e, g, j
Cs	mg/kg	2,35 ± 0,11	1,7E-01	1,8E-01	15	0,1	b, c, d
Cu	mg/kg	797± 13	2,2E+01	2,4E+01	23	0,1	b, c, e, g, i
Dy	mg/kg	2,730 ± 0,056	1,3E-01	6,7E-02	9	0,1	b, c, d
Er	mg/kg	1,495 ± 0,075	9,9E-02	9,9E-02	11	0,1	b, c, d
Eu	mg/kg	0,671 ± 0,021	4,3E-02	2,5E-02	9	0,1	b, c, d, j
Fe	%(m/m)	7,63 ± 0,14	1,6E-01	3,1E-01	29	0,1	b, c, e, h, i
Ga	mg/kg	14,58 ± 0,35	7,7E-01	5,6E-01	16	0,1	b, c, d, g

Constituent	Unit	Mass fraction	Repeatability standard deviation	Reproducibility standard deviation	No. of data sets	Minimum sample (g)[*1]	Analytical methods
Gd	mg/kg	3,08 ± 0,11	1,7E-01	1,4E-01	10	0,1	b, c, d
Hf	mg/kg	2,87 ± 0,11	2,1E-01	1,0E-01	6	0,1	d
Hg	mg/kg	5,06 ± 0,29	2,6E-01	3,7E-01	10	0,1	b, c, j
Ho	mg/kg	0,534 ± 0,031	4,4E-02	3,5E-02	8	0,1	c, d
In	mg/kg	15,20 ± 0,80	8,4E-01	1,2E+00	14	0,1	b, c, d
K	%(m/m)	0,862 ± 0,020	2,0E-02	3,5E-02	19	0,1	a, b, c, e, i
La	mg/kg	18,98 ± 0,79	1,2E+00	1,2E+00	15	0,1	b, c, d, j
Li	mg/kg	10,68± 0,90	5,3E-01	1,1E+00	10	0,1	b, c, e
Mg	%(m/m)	0,672 ± 0,012	1,5E-02	1,9E-02	16	0,1	c, e, i
Mn	%(m/m)	0,1843 ± 0,0026	5,7E-03	5,1E-03	24	0,1	c, e, i
Mo	mg/kg	2,96 ± 0,27	2,0E-01	3,8E-01	13	0,1	b, c, f
Nb	mg/kg	6,12 ± 0,54	6,0E-01	6,8E-01	10	0,1	b, c, d
Nd	mg/kg	16,32 ± 0,19	7,5E-01	2,1E-01	8	0,1	c, d
Ni	mg/kg	21,72 ± 0,96	1,4E+00	1,4E+00	13	0,1	b, c, e, g
P	mg/kg	480 ± 28	2,7E+01	4,2E+01	14	0,1	b, c, g, i
Pb	%(m/m)	1,212 ± 0,038	3,2E-02	6,9E-02	21	0,1	b, c, e, g, i
Pr	mg/kg	4,36 ± 0,11	2,3E-01	1,4E-01	10	0,1	b, c, d
Rb	mg/kg	45,2± 1,3	1,9E+00	2,2E+00	16	0,1	b, c, d
S	%(m/m)	6,87 ± 0,11	1,8E-01	2,1E-01	23	0,1	b, c, i, k, l
Sb	mg/kg	30,2 ± 1,9	2,1E+00	2,5E+00	11	0,1	b, d, f, g, j
Sc	mg/kg	5,22 ± 0,10	2,3E-01	8,9E-02	5	0,25	b, j
Si	%(m/m)	18,99 ± 0,38	2,5E-01	6,1E-01	16	0,1	c, e, g, h, i
Sm	mg/kg	3,238 ± 0,091	1,9E-01	1,2E-01	11	0,1	c, d, j
Sn	mg/kg	11,84 ± 0,78	1,0E+00	1,4E+00	19	0,1	b, c, d
Sr	mg/kg	402,0 ± 6,8	1,6E+01	1,3E+01	22	0,1	b, c, d, e
Tb	mg/kg	0,473 ± 0,019	2,3E-02	1,9E-02	6	0,1	d
Th	mg/kg	5,47 ± 0,24	3,4E-01	3,5E-01	13	0,1	b, c, d, j
Ti	%(m/m)	0,1977 ± 0,0040	5,5E-03	6,6E-03	17	0,1	e, i
Tm	mg/kg	0,212 ± 0,016	1,7E-02	1,9E-02	9	0,1	b, c, d
U	mg/kg	2,94 ± 0,11	1,6E-01	1,7E-01	16	0,1	b, c, d, j
V	mg/kg	169,2 ± 4,0	6,1E+00	5,9E+00	14	0,1	b, c, d, e, i
Y	mg/kg	14,83 ± 0,89	9,5E-01	1,4E+00	16	0,1	b, c, d, e, g
Yb	mg/kg	1,373 ± 0,053	7,7E-02	7,0E-02	11	0,1	b, c, d, j
Zn	%(m/m)	3,244 ± 0,065	6,7E-02	1,1E-01	18	0,2	a, c, e, i, j
Zr	mg/kg	104,0± 5,1	7,0E+00	7,3E+00	13	0,1	d, e, g, i

[*1]Minimum sample size used in the material characterization

ADDITIONAL INFORMATION ON COMPOSITION

Noncertified property values are provided for additional information only. Indicative values were derived from data that fulfilled the certification criteria although some sets of data were expressed with only one significant figure. An informative value is a value for which there is no sufficient information available to assess adequately the uncertainty associated.

Indicative Values

Constituent	Unit	Mass fraction	Repeatability standard deviation	Reproducibility standard deviation	No. of sets of data	Minimum sample (g)[*1]	Analytical methods
Se	mg/kg	5,98 ± 0,76	6,5E-01	1,1E+00	13	0,1	b, c
Ta	mg/kg	0,443 ± 0,068	6,1E-02	8,1E-02	9	0,1	b, d

[*1]Minimum sample size used in the material characterization

Informative Values

Constituent	Unit	Mass fraction	Range of sets of data average	No. of sets of data	Minimum sample (g)[*1]	Analytical methods
Au	mg/kg	0,062	0,058 - 0,066	4	1	j, m
B	mg/kg	41	30 - 55	4	0,02	c, f, g
F	%(m/m)	0,047	0,044 - 0,049	2	0,15	f
Ge	mg/kg	20	15 - 23	5	0,02	d, f, g
Lu	mg/kg	0,21	0,21 - 0,22	6	0,1	d
Na	%(m/m)	0,03	0,03 - 0,05	17	0,1	a, b, c, e, i, j
Re	mg/kg	0,003	0,001 - 0,006	5	0,25	b
Te	mg/kg	0,68	0,64 - 0,77	8	0,2	b
Tl	mg/kg	0,41	0,35 - 0,47	5	0,2	b, c
W	mg/kg	78	76 - 80	9	0,1	b, c, d

[*1]Minimum sample size used in the material characterization

An X-ray diffraction analysis identified quartz, gypsum, calcite and minerals from the mica, amphibole, chlorite, alunite and hydrotalcite supergroups /groups, probably close to muscovite, hornblende, chamosite, jarosite and hydrowoodwardite, respectively. Gypsum, calcite, jarosite and hydrowoodwardite are likely synthetic phases precipitated from metallurgical residues, rather than minerals, whereas quartz, hornblende, chlorite and mica might be refractory minerals withstanding the ore's processing.

INSTRUCTIONS FOR HANDLING AND USE

The material sachet must be opened only for sampling. The material must not be put in contact with other chemicals in order to avoid cross contamination.

The sample mass for analyses should not be less than the minimum sample size used in the material characterization. The property value and its associated uncertainty are only guaranteed if the minimum sample size is respected.

Analyses should be performed on samples as received. Separate samples should be weighed before and after drying at 105 ± 2 °C to a constant weight to obtain a correction factor for moisture.

STORAGE

The material must be stored in its original package, at room temperature in a clean and dry place.

HEALTH AND SAFETY INFORMATION

This material contains fine mineral particulate. Avoid dust dispersion, inhalation, eye contact or skin contact. Dispose of residual material in accordance with regulations pertaining for inorganic chemical and mineralogical waste.

METROLOGICAL TRACEABILITY

In the characterization process by an interlaboratory program, the selection of measurement methods as well as respective calibrants was based on the decision of each participating laboratory. A consequence of such an approach is that the metrological traceability chain(s) for each of the assigned values (combined from a number of results) cannot easily be described, but are expected to include independent sources of bias. Therefore, the demonstrated agreement of independent measurements resulting from the various methods, calibrants, and validation steps, using previously certified materials, results in certified values that are metrologically traceable to the SI units of mass and amount of substance.

ANALYTICAL METHODS

a acid digestion / flame atomic absorption spectrometry
b acid digestion / inductively coupled plasma mass spectrometry
c acid digestion / inductively coupled plasma optical emission spectrometry
d fusion / inductively coupled plasma mass spectrometry
e fusion / inductively coupled plasma optical emission spectrometry
f alternative current arc atomic emission spectrometry
g direct current arc atomic emission spectrometry
h fused pellet /energy dispersive X-ray fluorescence spectrometry
i fused pellet / wavelength dispersive X-ray fluorescence spectrometry
j instrumental neutron activation analysis
k combustion / infrared spectrometry
l combustion / gravimetry
m fire assay / inductively coupled plasma optical emission spectrometry

PARTICIPATING LABORATORIES

- Accurassay Laboratories, Thunder Bay, Canada
- Activation Laboratories Ltd., Ancaster, Canada
- AGAT Laboratories, Mississauga, Canada
- ALS Geochemistry Brisbane, Stafford, Australia
- ALS Geochemistry Vancouver, North Vancouver, Canada
- ALS Minerals Loughrea, Loughrea, Ireland
- Bureau Veritas Commodities Canada, Vancouver, Canada
- Central Geological Laboratory of Mongolia, Ulaanbaatar, Mongolia
- Eurotest Control EAD - Department Chemical Investigations, Sofia, Bulgaria
- hrltesting Pty Ltd, Assay Laboratory,Albion, Australia
- Intertek Laboratory Services, Wingfield, Australia
- Labwest Minerals Analysis Pty Ltd, Malaga, Australia
- Newcrest Laboratory Services, Orange, Auatralia
- MINTEK - Analytical Services Division, Randburg, South Africa
- SGS Canada Minerals Burnaby - Geochemistry, Burnaby, Canada
- SGS Canada Minerals Lakefield - Analytical, Lakefield, Canada
- Vinogradov Institute of Geochemistry of Siberian Branch of Russian Academy of Sciences - Laboratory of Optical Spectral Analysis and Certified Reference Materials, Irkutsk, Russia

PERIOD OF VALIDITY

The certified values are valid until December 2021, provided the RMMG-2 unit is handled and stored in accordance with instructions given in this certificate. This certification is nullified if the material is damaged, contaminated or otherwise modified. The stability of RMMG-2 will be monitored over the period of validity. Updates will be published at www.cetem.gov.br/crm.

FURTHER INFORMATION

The certification report is available upon request to CETEM. For details on the interpretation of measurement results on CETEM's certified reference materials, access the publication "Application Guide 1" at www.cetem.gov.br/crm.

CERTIFYING OFFICER

The technical and management aspects involved in the preparation, certification and issuance of RMMG-2 were coordinated through CETEM's Certified Reference Material Program.

Maria Alice Goes
Certified Reference Material Program Coordinator

REFERENCES

[1] ISO 5725-5:1998. Accuracy (trueness and precision) measurement methods and results – Part 5: Alternative methods for determination of the precision of a standard measurement method. International Organization for Standardization (ISO), Geneva.

[2] ISO 13528:2015 – Statistical methods for use in proficiency testing by interlaboratory comparisons. International Organization for Standardization (ISO), Geneva.