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CERTIFICATE OF ANALYSIS

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ACCREDITED
CERTIFICATE #4015.01

BXBA-4

Bauxite (Baixão de Ipiúna, Bahia)

Original certificate: April, 2014
Revision: July, 2018

The BXBA-4 is a crude bauxite sample originating from Baixão de Ipiúna region, located in Bahia State, Brazil. The raw material was oven-dried, crushed and pulverized to pass a 0.150 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. A unit of BXBA-4 consists of 100 g of powdered ore, packaged in a glass bottle.

This material was certified by means of an interlaboratory program involving thirty one expert laboratories in this field, using methods of their choice. Robust statistical methods [1] were performed on the data to estimate the property values and variability components. The certified values were assigned based on a minimum of five accepted sets of results, analytical methods suitable for the content of the constituent 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 combining the uncertainty components due to batch inhomogeneity and batch characterization [2].

Certified Values

Constituent	Unit	Mass fraction	Repeatability standard deviation [1]	Between-laboratory standard deviation [1]	No. sets of data	Minimum sample (g)[*3]	Analytical methods
Available alumina[*1]	% m/m	43.66 ± 0.34	1.5E-01	5.0E-01	15	0.65	i; k
Reactive silica[*2]	% m/m	4.53 ± 0.14	9.2E-02	1.9E-01	16	0.5	f; g; h; i; j
Al_2O_3	% m/m	49.75 ± 0.28	1.8E-01	5.6E-01	26	0.5	e; l; o; p; q
Fe_2O_3	% m/m	12.785 ± 0.076	8.6E-02	1.1E-01	22	0.1	l; n; p; r
SiO_2	% m/m	8.447 ± 0.083	5.7E-02	1.5E-01	24	0.1	b; n; p
TiO_2	% m/m	1.549 ± 0.027	2.2E-02	3.9E-02	27	0.1	c; l; n; p
ZrO_2	% m/m	0.0283 ± 0.0025	1.4E-03	3.9E-03	18	0.1	c; d; m; p; q
P_2O_5	% m/m	0.1952 ± 0.0032	2.1E-03	5.5E-03	20	0.5	l; p
V_2O_5	% m/m	0.0227 ± 0.0028	7.0E-04	4.4E-03	17	0.1	c; d; m; p; q
MnO_2	% m/m	0.0348 ± 0.0033	9.4E-04	5.9E-03	20	0.1	c; d; l; m; p
SO_3	% m/m	0.229± 0.012	6.0E-03	1.2E-02	10	0.5	c; p; s
Total organic carbon	% m/m	0.276 ± 0.064	6.3E-03	7.7E-02	9	0.1	s; t
Loss of mass 405 °C	% m/m	23.35 ± 0.17	6.3E-02	1.9E-01	11	0.5	u; v
Loss of mass 1000 °C	% m/m	27.27 ± 0.21	5.6E-02	1.8E-01	25	0.5	u; v

[*1]Amount of alumina that is digested in a caustic solution (150 °C) at similar conditions of Bayer Process.

[*2]Amount of silica that reacts with sodium hydroxide (150 °C) at similar conditions of Bayer Process.

[*3]Minimum test sample size used in the interlaboratory measurement program.

ADDITIONAL INFORMATION ON COMPOSITION

Noncertified property values are provided for additional information only. Indicative values were assigned based on a minimum of three accepted sets of results and analytical methods suitable for the content of the constituent. Informative values were estimated from a minimum of two accepted sets of results.

Indicative Values

Constituent	Unit	Mass fraction	Repeatability standard deviation [1]	Between-laboratory standard deviation [1]	No. of sets of data	Minimum sample (g)[*3]	Analytical methods
Cr_2O_3	% m/m	0.0010 ± 0.0003	2.1E-04	1.5E-04	4	0.15	a; c; d; r
CaO	% m/m	0.014 ± 0.006	9.6E-04	4.1E-03	11	0.5	p
MgO	% m/m	0.02 ± 0.01	3.3E-03	1.2E-02	13	0.5	c; l; p; q
Na_2O	% m/m	0.02 ± 0.01	3.6E-03	8.7E-03	6	0.7	p

[*3]Minimum test sample size used in the interlaboratory measurement program.

Informative Values

Constituent	Unit	Mass fraction	Range of sets of data average	No. of sets of data	Minimum sample (g)[*3]	Analytical methods
ZnO	% m/m	0.006	0.003 - 0.009	15	0.1	d; m; p
K_2O	% m/m	0.007	0.002 - 0.011	10	0.5	p
CuO	% m/m	0.003	0.001 - 0.006	4	0.1	a; d; m; p
Ga_2O_3	% m/m	0.007	0.005 - 0.008	6	0.5	p

[*3]Minimum test sample size used in the interlaboratory measurement program.

The mineral composition of BXBA-4 was identified by X-ray diffraction (XRD). The major mineral is gibbsite. Goethite, quartz and kaolinite were identified as minor minerals. Trace minerals include hematite, anatase, magnetite, ilmenite and zircon.

INSTRUCTIONS FOR USE

Analyses must be performed on sample that has been previously dried for at least 16 h in an oven controlled at 105 ± 2 °C. The contents of the bottle should be mixed (by rolling the bottle) before taking samples. The mass of samples used for analyses should be greater than the minimum size indicated for certified and indicative property values. Avoid prolonged exposure to air. Tightly recap the bottle after sampling.

STORAGE

The material should be stored at ambient temperature in a dry place.

HAZARDS SITUATION

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

LEVEL OF HOMOGENEITY

To assess homogeneity, twety units were selected from the batch of BXBA-4 using a stratified random sampling scheme. For each selected unit, measurements were carried out in triplicate, under repeatability conditions, by : fused pellet (1 g of sample) / X-ray fluorescence spectrometry (oxides), caustic digestion (1.3 g of sample) / titrimetry (available alumina); caustic digestion (1.3 g of sample) / flame atomic absorption spectrometry (reactive silica); caustic digestion (1.3 g of sample) – combustion at 680 °C – oxidation / infrared spectrometry (total organic carbon) and drying (1 g of sample) / thermal gravimetric analysis (loss of mass at 405 °C and at 1000 °C). A analysis of variance approach was performed on the data to compute the repetability and the between-unit standard deviations. For the content of SO_3, the uncertainty component due to batch inhomogeneity was estimated based on results obtained from the BXMG-1 homogeneity study [3]. The uncertainty component due to batch inhomogeneity, expressed as a percentage of the certified value, is less than 2 %.

LEVEL OF STABILITY

BXBA-4 is considered to be stable. Based on the nature of the material and previous chemical and mineralogical analysis, the degradation is not anticipated provided the material is handled and stored in accordance with instructions given in this certificate.

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 alternative arc discharge atomic emission spectrometry
b acid digestion / gravimetry
c acid digestion / inductively coupled plasma optical emission spectrometry
d acid digestion / inductively coupled plasma mass spectrometry
e acid digestion / titrimetry
f caustic digestion / flame atomic absorption spectrometry
g caustic digestion / flame emission spectrometry
h caustic digestion / gravimetry
i caustic digestion / inductively coupled plasma - optical emission spectrometry
j caustic digestion / spectrophotometry
k caustic digestion / titrimetry
l fusion / flame atomic absorption spectrometry
m fusion / inductively coupled plasma mass spectrometry
n fusion / spectrophotometry
o fusion / titrimetry
p fused pellet / X-ray fluorescence spectrometry
q pressed pellet / X-ray fluorescence spectrometry
r instrumental neutron activation analysis
s combustion / infrared spectrometry
t oxidation / infrared spectrometry
u calcination / gravimetry
v thermal gravimetric analysis

PARTICIPATING LABORATORIES

- Acme Analytical Laboratories Ltd., Vancouver, Canada
- Activation Laboratories Ltd., Ancaster, Canada
- Alcoa Alumínio S/A - Fábrica de Alumínio, Laboratório, Andradas, Brasil Alcoa Minerals of Jamaica - Laboratory Department, Kingston, Jamaica
- Alcoa Minerals of Jamaica - Laboratory Department, Kingston, Jamaica
- Alcoa Productos Primarios Europa - Laboratory Department, San Ciprian, Spain
- Alcoa World Alumina Australia - Pinjarra Laboratory, Pinjarra, Western Australia
- Alcoa World Alumina Australia - Kwinana Mining Laboratory, Kwinana, Western Australia
- Alcoa World Alumina Brasil Ltda - Mina de Bauxita de Juruti, Juruti, Brasil
- Alcoa World Alumina - Technology Delivery Group, Kwinana, Western Australia
- Alcoa World Alumina Atlantic - Point Comfort Operations, Point Comfort, Unites States of America
- ALS Minerals - Geochemistry Division, North Vancouver, Canada
- ALTEO Gardanne, Gardanne Cedex, France
- Central Geological Laboratory of Mongolia, Ulaanbaatar, Mongolia

- Centro de Tecnologia Mineral - Coordenação de Análises Minerais, Rio de Janeiro, Brasil
- Comisión Chilena de Energía Nuclear – Centro de Estudios Nucleares La Reina - Laboratorio Análisis por Activación Neutrónica, Santiago, Chile
- Comissão Nacional de Energia Nuclear - Instituto de Radioproteção e Dosimetria, Rio de Janeiro, Brasil
- Companhia Brasileira de Alumínio – Laboratório Químico, Alumínio, Brasil
- Consórcio de Alumínio do Maranhão, São Luis, Brasil
- Escola Politécnica da USP – Departamento de Engenharia de Minas e de Petróleo – Laboratório de Caracterização Tecnológica, São Paulo, Brasil
- Eurotest Control EAD – Department Chemical Investigations, Sofia, Bulgaria
- Hydro Alumina do Norte do Brasil S/A, Laboratório, Barcarena, Brasil
- Intertek Brazil Minerals, Cotia, Brasil
- Institute of Geochemistry Siberian Branch of Russian Academy of Sciences - Laboratory of Optical Spectral Analysis and Certified Reference Materials, Irkutsk, Russia
- Instituto de Tecnología Cerámica, Laboratorio de Análisis Químico, Castellón, Spain
- L.A. Teixeira & Filho S/C Ltda, Andradas, Brasil
- Mineração Rio do Norte – Laboratório Químico, Porto Trombetas, Brasil
- Rio Tinto Alcan Business Development & Growth Technology and R&D, Pullenvale, Australia
- Rio Tinto Alcan Weipa – Weipa Bauxite Laboratory, Weipa, Australia
- Rio Tinto Aluminium - Yarwun Refinery, Queesland, Australia
- Serviço Geológico Minero Argentino, Buenos Aires, Argentina
- Suriname Aluminum Company - Laboratories, Paramaribo, Suriname

PERIOD OF VALIDITY

The certified values are valid until April 2034, provided the BXBA-4 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 BXBA-4 will be monitored over the period of validity. Updates will be published on the CETEM website.

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/mrc.

CERTIFYING OFFICER

The technical and management aspects involved in the preparation, certification and issuance of the BXBA-4 were coordinated through the 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 Guide 35:2006. Reference materials – General and statistical principles for certification. International Organization for Standardization (ISO), Geneva.

[3] PMRC 2010.03.03 – BXMG-1 Certification Report. Certified Reference Material Program, CETEM. Rio de Janeiro.