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CERTIFICATE OF ANALYSIS

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ACCREDITED
CERTIFICATE #4015.01

BXPA-3

Bauxite (Paragominas, Pará)

Original certificate: July, 2010
Revision: July, 2018

The BXPA-3 is a washed bauxite sample originating from Paragominas region, located in Pará 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 BXPA-3 consists of 105 g of powdered ore, packaged in a glass bottle.

This material was certified by means of an interlaboratory program involving thirty five 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 assignment of certified property values was determined by the quality of the data based on such aspects as a minimum of ten accepted sets of data, the analytical methods used, the uncertainty associated with each property value and fitness for 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	49.76 ± 0.31	2.9E-01	5.4E-01	22	0.5	j; l
Reactive silica[*2]	% m/m	3.42 ± 0.25	1.1E-01	3.8E-01	21	0.5	g; h; i; j; k
Al_2O_3	% m/m	53.73 ± 0.37	2.6E-01	6.9E-01	32	0.1	c; f; s; w
Fe_2O_3	% m/m	11.63 ± 0.14	7.0E-02	3.1E-01	36	0.1	c; f; r; s; v; w
SiO_2	% m/m	4.187 ± 0.095	3.3E-02	1.4E-01	29	0.1	b; c; s; w
TiO_2	% m/m	1.924 ± 0.030	1.5E-02	6.0E-02	29	0.1	e; s; u; v; w
ZrO_2	% m/m	0.207 ± 0.016	2.7E-03	2.3E-02	15	0.1	c; s; t; v; w
P_2O_5	% m/m	0.0423 ± 0.0038	1.3E-03	2.5E-03	16	0.1	c; s; w
V_2O_5	% m/m	0.0293 ± 0.0011	1.3E-03	1.5E-03	14	0.1	c; s; t; v; w
Cr_2O_3	% m/m	0.0155 ± 0.0019	6.0E-04	2.6E-03	17	0.1	a; c; s; t; v; w
MnO_2	% m/m	0.0454 ± 0.0019	1.0E-03	3.2E-03	20	0.1	a; c; r; s; t; v; w
Loss of mass 1000 °C	% m/m	28.19 ± 0.27	5.2E-02	1.8E-01	21	0.05	m; n
Loss of mass 1075 °C	% m/m	28.28 ± 0.28	6.2E-02	1.2E-01	8	1	m

[*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]smallest mass sample used in the interlaboratory measurement program.

ADDITIONAL INFORMATION ON COMPOSITION

Noncertified property values are provided for information only. Indicative values were assigned to property values derived from at least eight sets of data that did not fulfill a specific statistical criteria required for certification, but which uncertainty is fit-for-purpose. Informative values were estimated from a minimum of three sets of data.

Indicative Values

Constituent	Unit	Mass fraction	Repeatability standard deviation [1]	Between-laboratory standard deviation [1]	No. of sets of data	Minimum sample (g)[*1]	Analytical methods
CaO	% m/m	0.01 ± 0.01	4.7E-04	1.3E-02	13	0.1	w
MgO	% m/m	0.03 ± 0.02	3.5E-03	2.5E-02	12	0.1	s; w
ZnO	% m/m	0.003 ± 0.001	4.8E-04	1.5E-03	13	0.1	c; d; q; s; w
K_2O	% m/m	0.011 ± 0.004	5.4E-04	4.2E-03	12	0.1	a; w
SO_3	% m/m	0.09 ± 0.04	2.4E-03	4.1E-02	8	0.1	o; p; w

[*1]smallest mass sample used in the interlaboratory measurement program.

Informative Values

Constituent	Unit	Mass fraction	Range of sets of data average	No. of sets of data	Analytical methods
Na_2O	% m/m	0.02	0.011 - 0.03	4	v; w
CuO	% m/m	0.001	0.0002 - 0.003	4	a; d; q; s
Ga_2O_3	% m/m	0.010	0.008 - 0.016	4	q; t; w
Total organic carbon	% m/m	0.06	0.04 - 0.07	5	p

The mineral composition of BXPA-3 was identified by X-ray diffraction (XRD). The major mineral is gibbsite. Hematite was identified as minor mineral. Kaolinite, goethite, anatase, quartz, boehmite, zircon, diaspore, ilmenite and muscovite were detected as trace minerals.

INSTRUCTIONS FOR USE

Analyses must be performed on samples that have 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.

HAZARDOUS SITUATION

This material contains fine mineral particulate. Avoid dispersion of, exposure to dust by 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, ten units were selected from the batch of BXPA-3 using a stratified random sampling scheme. For each selected unit, measurements were carried out in triplicate, under repeatability conditions, by fused pellet (1.23 g of sample) / X-ray fluorescence spectrometry (oxides), caustic digestion (0.65 g of sample) / titrimetry (available alumina) and caustic digestion (0.65 g of sample) / inductively coupled plasma optical emission spectrometry (reactive silica). A one-way analysis of variance approach was performed on the data to compute the within and the between-unit standard deviations. The uncertainty component due to batch inhomogeneity, expressed as a percentage of the certified value, is less than 4 %.

LEVEL OF STABILITY

BXPA-3 is considered to be stable. Based on the nature of the material and previous chemical and mineralogical analysis, deterioration 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 acid digestion / flame atomic absorption 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 / spectrophotometry
f acid digestion / titrimetry
g caustic digestion / flame atomic absorption spectrometry
h caustic digestion / flame photometry
i caustic digestion / gravimetry
j caustic digestion / inductively coupled plasma optical emission spectrometry
k caustic digestion / spectrophotometry
l caustic digestion / titrimetry
m calcination / gravimetry
n calcination / thermal gravimetric analysis
o combustion / gravimetry
p combustion / infrared spectrometry
q direct current atomic emission spectrometry
r fusion / flame atomic absorption spectrometry
s fusion / inductively coupled plasma optical emission spectrometry
t fusion / inductively coupled plasma mass spectrometry
u fusion / spectrophotometry
v instrumental neutron activation analysis
w fused pellet / X-ray fluorescence spectrometry

PARTICIPATING LABORATORIES

- Acme Analytical Laboratories Ltd., Vancouver, Canada
- Activation Laboratories Ltd., Ancaster, Canada
- Alcoa Alumínio S/A - Fábrica de Alumínio, Laboratório, Poços de Caldas, Brasil
- Alcoa World Alumina Australia - Pinjarra Laboratory, Pinjarra, Western Australia
- Alcoa World Alumina Australia - Kwinana Mining Laboratory, Kwinana, Western Australia
- Alcoa Productos Primários Europa - Laboratory Department, San Ciprian, Spain
- Alcoa World Alumina - Technology Delivery Group, Kwinana, Western Australia
- Alcoa World Alumina Atlantic - Point Comfort Operations, Point Comfort, Unites States of America
- ALS Chemex, North Vancouver, Canada
- Alumina do Norte do Brasil S/A, Laboratório, Barcarena, Brasil
- Anglo Research - Crown Mines, Johannesburg, South Africa
- Bundesanstalt für Geowissenschaften und Rohstoffe - Geochemie, Hannover, Germany
- Central Geological Laboratory, Ulaanbaatar, Mongolia

- Comissão Nacional de Energia Nuclear - Centro de Desenvolvimento da Tecnologia Nuclear, Laboratório Químico, Belo Horizonte, Brasil
- Centro de Tecnologia Mineral - Coordenação de Análises Minerais, Rio de Janeiro, Brasil
- Companhia Brasileira de Alumínio – Laboratório Químico, Alumínio, Brasil
- Comisión Chilena de Energía Nuclear - Laboratorio de Análisis por Activación Neutrónica, Santiago, Chile
- Consórcio de Alumínio do Maranhão, São Luis, Brasil
- Council for Geoscience, Geochemistry and Laboratory Units, Pretoria, South Africa
- Eurotest Control JSC, Sofia, Bulgaria
- Geological Survey of Israel, Division of Geochemistry and Environmental Geology, Jerusalem, Israel
- Geoscience Laboratories, Sudbury, Canada
- Instituto de Tecnología Cerámica, Chemical Analysis Unit, Castellón, Spain
- Institute of Geochemistry Siberian Branch of Russian Academy of Sciences - Laboratory of Optical Spectral Analysis and Certified Reference Materials, Irkutsk, Russia
- Jamalco - Laboratory Department, Kingston, Jamaica
- L.A. Teixeira & Filho, Andradas, Brasil
- Mineração Curimbaba Ltda, Laboratório, Poços de Caldas, Brasil
- Mineração Rio do Norte – Laboratório Químico, Oriximiná, Brasil
- MINTEK - Analytical Services Division, Randburg, South Africa
- Novelis do Brasil Ltda - Laboratório Químico, Ouro Preto, Brasil
- Rio Tinto Alcan - Queensland Research & Development Centre, Pullenvale, Australia
- Serviço Geológico Minero Argentino – Laboratório, Buenos Aires, Argentina
- Suriname Aluminum Company - Laboratories, Paramaribo, Suriname
- Vale – Departamento de Desenvolvimento de Projetos Minerais - Laboratório, Santa Luzia, Brasil
- Vale Paragominas – Laboratório Químico, Paragominas, Brasil

PERIOD OF VALIDITY

The certified values are valid until July 2032, provided the BXPA-3 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 BXPA-3 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 BXPA-3 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.