

CERTIFICATE OF ANALYSIS

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BXPA-1Bauxite (Jurutí, Pará)

Original certificate: October, 2006 Revision: July, 2018

The BXPA-1 is a washed bauxite sample originating from Jurutí region, located in Pará State, Brazil. The raw material was oven-dried, crushed 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. A unit of BXPA-1 consists of 120 g of powdered ore, packaged in a glass bottle.

This material was certified by means of an interlaboratory program involving twenty seven expert laboratories in this field, using methods of their choice. A one-way analysis of variance technique [1] were performed on the data to estimate the overall mean 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, ratio of the betweento the within variability component less than or equal to 3 and percentage of sets that are rejected do not exceed 15% [2]. The stated uncertainty is an expanded uncertainty, with coverage factor 2, estimated by combining the uncertainty components due to due to batch inhomogeneity and batch characterization [3].

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	49.0± 0.3	1.9E-01	5.0E-01	12	0.65	j; g
Reactive silica*2	% m/m	3.8 ± 0.2	1.4E-01	2.2E-01	11	0.65	c; e; m; p; q; s
Al_2O_3	% m/m	52.8 ± 0.4	4.6E-01	5.7E-01	22	0.1	c; e; q; m; p; s
Fe ₂ O ₃	% m/m	12.8 ± 0.1	1.1E-01	2.4E-01	27	0.1	a; c; e; q; m; p; r; s
TiO ₂	% m/m	1.42 ± 0.02	1.9E-02	4.4E-02	22	0.1	c; d; q; l; m; r; s
V ₂ O ₅	% m/m	0.058 ± 0.005	3.5E-03	8.9E-03	14	0.1	c; q; l; m; r; s
ZnO	% m/m	0.004 ± 0.001	9.9E-04	2.0E-03	9	0.2	a; c; m; p; s
Loss of mass 1000 °C	% m/m	27.5 ± 0.4	1.3E-01	2.9E-01	20	1.0	k

 $^{^{*1}}$ amount of alumina that is digested in a caustic solution (150 $\,^{\circ}$ 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 for BXPA-1 characterization.

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.

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
SiO ₂	% m/m	4.93 ± 0.05	4.1E-02	8.1E-02	15	0.1	b; m;o;s
ZrO ₂	% m/m	0.066 ± 0.005	3.1E-03	8.7E-03	13	0.1	q; m; r; s
P ₂ O ₅	% m/m	0.018 ± 0.004	2.4E-03	6.2E-03	10	0.2	c; d; m; n; s
MnO	% m/m	0.0017 ± 0.0004	6.1E-04	4.8E-04	9	0.2	c; d; q; s

^{*1} smallest mass sample used in the interlaboratory measurement program for BXPA-1 characterization

The mineral composition of BXPA-1 was identified by X-ray diffraction (XRD). The major mineral is gibbsite. Kaolinite. goethite and hematite were identified as minor minerals. 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-1 using a stratified random sampling scheme. For each selected unit, measurements were carried out in triplicate, under repeatability conditions, by fused pellet (0.5 g of sample) / X-ray fluorescence spectrometry (oxides). A one-way analysis of variance approach was performed on the data to compute the within and the between-unit standard deviations. For available alumina, reactive silica and loss of mass (1000 °C), the uncertainty components due to batch inhomogeneity were estimated based on data obtained, respectively, from the BXPA-3, BXPA-4 and BXMG-2 homogeneity studies. The uncertainty component due to due to batch inhomogeneity, expressed as a percentage of the certified value, is less than 2 %.

LEVEL OF STABILITY

BXPA-1 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 / visible UV spectrometry
- e acid digestion / titrimetry
- f caustic digestion / flame atomic absorption spectrometry
- g caustic digestion / inductively coupled plasma optical emission spectrometry
- h caustic digestion / visible UV spectrometry
- i caustic digestion / gravimetry
- j caustic digestion / titrimetry
- k calcination / gravimetry
- I fusion / flame atomic absorption spectrometry
- m fusion / inductively coupled plasma optical emission spectrometry
- n fusion / visible UV spectrometry
- o fusion / gravimetry
- p fusion / titrimetry
- q instrumental neutron activation analysis
- r pressed pellet / X-ray fluorescence spectrometry
- s fused pellet / X-ray fluorescence spectrometry

PARTICIPATING LABORATORIES

- Acme Analytical Laboratories Ltd., Vancouver, Canada
- Alcan Arvida R&D Center. Jonquière. 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 World Alumina Technology Delivery Group. Kwinana. Western Australia
- Alumina do Norte do Brasil S/A. Laboratório. Barcarena. Brasil
- Canada Center for Mineral and Energy Technology Mining and Mineral Sciences Laboratories Analytical Service Group. Ottawa. Canada
- Centro de Investigaciones para la Industria Minero-Metalúrgica Caracterización de Materiales. Ciudad de la Habana. Cuba
- Centro de Tecnologia Mineral Coordenação de Análises Minerais. Rio de Janeiro. Brasil
- Comisión Chilena de Energía Nuclear Laboratorio de Análisis por Activación Neutrónica. Santiago. Chile
- Comissão Nacional de Energia Nuclear Centro de Desenvolvimento da Tecnologia Nuclear. Laboratório Químico. Belo Horizonte. Brasil
- Companhia Brasileira de Alumínio Laboratório Químico, Alumínio, Brasil
- Consórcio de Alumínio do Maranhão. São Luis. Brasil
- Innolab do Brasil. Rio de Janeiro. Brasil
- Instituto de Geociências da Universidade Estadual de Campinas. Campinas. Brasil
- Instituto de Geociências da Universidade Federal do Rio de Janeiro. Rio de Janeiro. Brasil
- Instituto de Pesquisas Energéticas e Nucleares. São Paulo. Brasil
- Instituto de Radioproteção e Dosimetria Laboratório. Rio de Janeiro. Brasil



- L.A. Teixeira & Filho. Andradas. 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
- Serviço Geológico Minero Argentino Laboratório. Buenos Aires. Argentina
- SGS Geosol Laboratórios. Belo Horizonte. Brasil
- Vale Departamento de Desenvolvimento de Projetos Minerais Laboratório. Santa Luzia. Brasil
- Treibacher Schleifmittel Brazil. Salto. Brasil

PERIOD OF VALIDITY

The certified values are valid until October 2028, provided the BXPA-1 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-1 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-1 were coordinated through the CETEM's Certified Reference Material Program.

Maria Alice Goes Certified Reference Material Program Coordinator

REFERENCES

- [1] ISO 5725-2:1998. Accuracy (trueness and precision) measurement methods and results Part 2: Basic method for determination of repeatability and reproducibility of a standard measurement method. International Organization for Standardization (ISO). Geneva.
- [2] Steger. H.F. "A re-assessment of the criteria for certification in CCRMP"; Geostandards Newsletter 6:1: 17-23; 1982.
- [3] ISO Guide 35:2006. Reference materials General and statistical principles for certification. International Organization for Standardization (ISO). Geneva.