

Preliminary

Certificate

Microanalytical Reference Material

Apatite-NP-B01

Nano-particulate pressed powder pellet

Certified Values

Analyte	Value	Unc. (95% CL)	Unit	Method
Ca	38.8	0.5	g/100g	C, D
P	17.7	0.6	g/100g	A, C, D
Na	756	42	μg/g	A, C
Mg	221	15	μg/g	A, C
Al	246	44	μg/g	C, D
Si	5700	354	μg/g	C, D
S	1169	194	μg/g	A
V	16.1	1.8	μg/g	A, B
Mn	451	18	μg/g	A, C, D
Cu	5.7	1.2	μg/g	A
Ga	7.9	0.5	μg/g	A, B
As	52.3	2.4	μg/g	A
Sr	2705	68	μg/g	A, B, C, D
Y	243	7	μg/g	A, B
Nb	0.4	0.1	μg/g	A, B
Mo	0.3	0.1	μg/g	A
Cd	0.47	0.13	μg/g	A
Ba	23.8	1.8	μg/g	A, B

Certified Values

Analyte	Value	Unc. (95% CL)	Unit	Method
La	552	40	μg/g	A, B
Ce	1468	44	μg/g	A, B
Pr	187	8	μg/g	A, B
Nd	716	23	μg/g	A, B
Sm	112	4	μg/g	A, B
Eu	21.2	0.9	μg/g	A, B
Gd	72.0	3.5	μg/g	A, B
Tb	8.69	0.36	μg/g	A, B
Dy	44.9	1.5	μg/g	A, B
Ho	8.40	0.37	μg/g	A, B
Er	21.8	0.9	μg/g	A, B
Tm	2.99	0.16	μg/g	A, B
Yb	17.6	0.9	μg/g	A, B
Lu	2.52	0.14	μg/g	A, B
Pb	14.3	0.9	μg/g	A
Bi	0.35	0.04	μg/g	A
Th	101	6	μg/g	A, B
U	31.9	2.2	μg/g	A, B

Information Values

Analyte	Value	Unc. (95% CL)	Unit	Method
Li	1.1		μg/g	A
Sc	0.3		μg/g	A
Cr	7		μg/g	A
Fe	477	108	μg/g	A, C, D
Co	1.4		μg/g	A
Ni	1.7	1.1	μg/g	A
Ge	1.5		μg/g	A
Se	1.4		μg/g	A
Rb	0.2		μg/g	A, B

Information Values

Analyte	Value	Unc. (95% CL)	Unit	Method
Zr	10.7	5.5	μg/g	B
Ag	0.043		μg/g	A
In	0.007		μg/g	A
Sb	0.11		μg/g	A
Hf	0.1		μg/g	B
W	1.1		μg/g	A, B
Re	0.004		μg/g	A
LOI	2.46	0.04	g/100g	

The assigned values represent the mean of laboratory means. The reference values were obtained through measurements performed on the nano-powder using methods A, B, C and or D in two competent laboratories (ISO 17025^[1] accredited) based on the characterisation approaches “Characterisation of a non-operationally defined measurand using two or more methods of demonstrable accuracy in one or more competent laboratories” or “Characterization using a single reference measurement procedure (as defined in ISO/IEC Guide 99^[2]) in two laboratories”.

List of analytical methods used for certification:

- A Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) or Plasma-Atomic Emission Spectroscopy (ICP-AES) analysis following Four Acid Digestion
- B Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) analysis following Lithium Borate Fusion
- C Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) analysis following Lithium Borate Fusion
- D X-Ray Fluorescence Spectroscopy (XRF) analysis on Lithium Borate Fusion beads

Further, each Laboratory's performance was statistically evaluated following recommendations from ISO Guide 35^[3].

Homogeneity and stability tests were performed on the Nano-Pellets using LA-ICP-MS and in accordance with ASTM E826-14^[4], ISO 13528^[5], and ISO Guide 35.

The expanded uncertainty is composed of the uncertainty components from characterisation, as well as from the homogeneity, and stability. Unless stated otherwise a coverage factor $k = 2$ was applied to reach a confidence level of 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement (GUM)^[6].

Information values did not fulfil all necessary statistical criteria of a reference value and should neither be considered for calibration nor validation. For most information values a homogeneity and stability test using LA-ICP-MS was not possible, due to concentrations being \leq limit of detection. The information values, which have an uncertainty were demoted due to the calculated combined expanded uncertainty not being usable.

The percent loss on ignition (LOI) is calculated from the gravimetrically measured differences in weight after decomposition of the samples in the furnace.

Preliminary Uranium-Lead Isotope Measurement Information Values (ID-TIMS)

Analyte	Value	Uncertainty (2SD)
²⁰⁶ Pb/ ²⁰⁴ Pb	36.02	0.41
²⁰⁶ Pb/ ²³⁸ U	0.09198	0.00103
²⁰⁷ Pb/ ²³⁵ U	0.7721	0.0383
²⁰⁷ Pb/ ²⁰⁶ Pb	0.06090	0.00263
Th/U	2.78	0.02
Pb _{radiogenic} /Pb _{common}	0.49	0.01
²⁰⁶ Pb/ ²³⁸ U Date	567.2 Ma	3.4 Ma (MSWD = 0.32; n = 10)

U-Pb date is calculated assuming an initial Pb isotopic composition equivalent to the Stacey and Kramers (1975)^[7] two-stage Pb evolution model (at 550 Ma) for the remainder of the common Pb, assigning an uncertainty component of 1 % to the model isotopic ratio.

Preliminary Strontium Isotope Measurement Information Values (ID-TIMS)

Analyte	Value	Uncertainty (2SD)	Uncertainty (95% CL)
$^{87}\text{Sr}/^{86}\text{Sr}$	0.704280	0.000026	0.000040

The $^{87}\text{Sr}/^{86}\text{Sr}$ isotope ratio was determined using an accredited method in which three aliquots were measured by TIMS after acid digestion and strontium separation.

The isotope measurement information values are preliminary. The final values will be given as soon as the homogeneity and stability tests are completed.

Pellet serial number: {SERIENNUMMER}

Date of dispatch: {LIEFERDATUM}

This certificate was approved by the myStandards GmbH, Kiel, 21.02.2023

Signed:

Simon Nordstad
CEO & Founder
Method Development & Production

Christina Wittke
CEO & Founder
Management & Marketing

Intended Use

This microanalytical certified reference material (CRM) is designed for use by laboratories undertaking the determination of major and trace element mass fractions in apatite and equivalent matrices with LA-ICP-MS (Laser Ablation Inductively Coupled Plasma Mass Spectrometry). It is suitable for calibration and as a secondary reference material for the assessment of a measurement procedure and quality control. Note that the material may only be used for a single purpose in the same measurement process. For example, it must not be used for calibration and method validation at the same time.

Description of the CRM

This CRM is a nanoparticulate pressed powder pellet of the apatite powder "Apatite-NP-B01". The original apatite crystals, originate from the Arusha Loliondo district in Tanzania, Africa. The crystals were crushed to a particle size of 63 μm . The resulting powder was subjected to our own material-specific milling protocol, freeze-dried, homogenised, and split into batches. Batch number 1 was pressed, into 10 mm diameter pellets, without any binders using a programmable hydraulic press. The fortification of contrasting colour surrounding the reference material is, according to the manufacturer, an "organic compound". The exact composition is not specified any closer. The certificate of analysis is available on demand.

Handling advice and Storage information

Avoid touching the pellet's surface directly in order to prevent contamination. Also, do not clean the surface with any liquids as it may compromise the pellet's integrity.

Please note the label marks the bottom of the pellet.

Store the CRM in a desiccator and or in a dark and dry environment.

The myStandards GmbH cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially with respect to opened samples.

Period of Validity

Provided the storage and handling conditions are met, no chemical alteration is known to exist, and the assigned values will remain stable. Therefore, the product information and assigned values for this CRM are valid for one year, pending stability monitoring, from the date of dispatch. This validity may be extended as further evidence of stability becomes available. The manufacturer will inform the customer if any alterations occur.

Safety instructions

Nano-particulate powders can cause harm if ingested, inhaled or in contact with skin. In their pressed form however, they do not exhibit any dusting. If a pellet should accidentally break, we advise wearing a dust mask during clean up.

Minimal sample size

The minimal sample size corresponds to a spot size of 50 μm.

Further ablation conditions and signal acquisition parameters during homogeneity- and stability-testing were:

<i>Laser fluence</i>	<i>6 J/cm²</i>
<i>Repetition rate</i>	<i>10 Hz</i>
<i>Background</i>	<i>20 s</i>
<i>Signal acquisition</i>	<i>30 s</i>

Metrological Traceability

This CRM has been produced in accordance with the recommendations specified in ISO 17034^[8] and ISO Guide 35 and are traceable to the base units of SI via calibrated measurements in two accredited laboratories (ISO 17025) or via calibrated measurements using a single reference measurement procedure in two accredited laboratories (ISO 17025). The preliminary values for uranium–lead isotope measurements are traceable to the units of SI via calibrated measurements using a single reference measurement procedure in a single laboratory^{[9][10]}. The preliminary value for strontium isotope measurements is also traceable to the units of SI via calibrated measurements using a single reference measurement procedure in an accredited laboratory (ISO 17025).

Further Information

A detailed report on the certification of the reference material is available on request or can be downloaded from www.my-standards.com.

Sample handling prior to pelletising is performed manually. Therefore, small amounts of the sample material may be seen on the fortification, this does not reduce the integrity of the CRM. Please refrain from using this part of the pellet.

The pellets are sold exclusively via the myStandards GmbH and authorised subcontractors.

Document History

<i>Version</i>	<i>Date</i>	<i>Changes applied</i>
1.0	11.02.2022	First publication
2.0	21.02.2023	Changed Description of Assigned Values and Metrological Traceability incl. updated References; Addition of the LOI; Addition of List of analytical methods used for certification; Updated link to terms and conditions; Adaptation to automatically fill in the date and individual pellet characteristics

References

- [1] **EN ISO/IEC 17025:2017 (D/E)**, *General requirements for the competence of testing and calibration laboratories*
- [2] **ISO/IEC Guide 99:2007**, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*
- [3] **ISO Guide 35:2017 (E)**, *Reference materials – Guidance for characterization and assessment of homogeneity and stability*
- [4] **ASTM E826-14**, *Standard Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectroscopy*. ASTM International, West Conshohocken, PA, 2014. www.astm.org
- [5] **ISO 13528:2015 (E)**, *Statistical methods for use in proficiency testing by interlaboratory comparison*
- [6] **ISO/IEC Guide 98-3:2008**, *Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*
- [7] **J.S. Stacey, J.D. Kramers (1975)**: *Approximation of terrestrial lead isotope evolution by a two-stage model*. Earth and Planetary Science Letters, 26, 207-221
- [8] **EN ISO 17034:2016 (D/E)**, *General requirements for the competence of reference material producers*
- [9] **D.J. Condon, B. Schoene, N.M. McLean, S.A. Bowring, R.R. Parrish (2015)**: *Metrology and traceability of U-Pb isotope dilution geochronology (EARTHTIME Tracer Calibration Part I)*. Geochimica et Cosmochimica Acta, Volume 164, 464-480
- [10] **N.M. McLean, D.J. Condon, B. Schoene, S. A. Bowring (2015)**: *Evaluating uncertainties in the calibration of isotopic reference materials and multi-element isotopic tracers (EARTHTIME Tracer Calibration Part II)*. Geochimica et Cosmochimica Acta, Volume 164, 481-501

Legal notice

Our order, sales and delivery conditions apply. The valid version of our general terms and conditions (status 01.09.2019) - can be found on our website: <https://www.my-standards.com/terms-and-conditions/>. They are also available on request.