CERTIFICATE OF ANALYSIS

FLX-136

Limestone

New certificate issued April 2022

Certified Values

	Mass fraction in %[1)]	Uncertainty[2)]	Traceable to
Al_2O_3	0,181	0,008	SI unit kg/kg
CaO	55,06	0,34	SI unit kg/kg
Fe_2O_3	0,126	0,010	SI unit kg/kg
K_2O[3)]	0,051	0,017	BCS-CRM No. 393
MgO	0,377	0,061	SI unit kg/kg
Mn_2O_3	0,038	0,010	SI unit kg/kg
SiO_2	0,548	0,053	SI unit kg/kg
SO_3	0,033	0,011	BCS-CRM No. 393
SrO	0,022	0,013	BCS-CRM No. 393

1) Certified value traceable to SI unit kg/kg based on **dried material**
2) Expanded uncertainty U_{CRM} calculated for a confidence interval of 95% (k=2) based on uncertainty of characterization.
3) The certified values for K2O are void when the material is ignited.

The sum of all oxides is 56,48 %. This includes informational values and excludes LOI.

This certificate is valid, within the uncertainty specified, **until 21.01.2030,** provided the CRM is handled in accordance with instructions given in this certificate. The certification is nullified if the CRM is damaged, contaminated, or otherwise modified.

Bedburg-Hau, **05.04.2022**

Responsible Reference Materials
Susan Aschenbrenner

General Manager
Dr. Rainer Schramm

Description of the CRM

This reference material sample was produced from commercial products. Material was taken directly from the production stream.
The complete batch was sealed into 50ml bottles.

Intended use

Calibration and control sample for x-ray fluorescence (XRF) analysis.

Informational Values

	Mass Fraction in %[3)]	Uncertainty[4)]
Na_2O	0,015	
P_2O_5	0,011	
TiO_2	0,012	0,019
ZnO	0,003	0,007
LOI	43,572	

3) Only Informational Value.
4) Expanded uncertainty U_{CRM} calculated for a confidence interval of 95% (k=2) based on uncertainty of characterization, if present.

Instructions for the correct use of the CRM

This material is moisture sensitive. It can only be used without any sample pretreatment if the original seal is intact. After opening the material has to be sealed again as soon as possible to avoid any change.

The minimum sample quantity for analysis should be 1.0g to be in agreement with the stated uncertainties.

For XRF use, samples should be prepared as a fused bead, e.g. in accordance with DIN 51001.

If the sample is used after ignition (1050°C to constant mass), the ignited certified values have to be calculated, using the following formula:

$conc_{ignited} = \frac{conc_{original} * Sum}{Sum - LOI}$, while the LOI must be determined.

Attention: The certified values for K_2O are void, as soon as the material is ignited.

Storage Information

The material has to be stored in a dry and clean environment.

Hazardous situation

For this material an actual MSDS is available.

Level of homogeneity

In accordance with ISO Guide 35:2017 a homogeneity study was performed. A one-way ANOVA was used to calculate the batch inhomogeneity u_{bb}^2.

$$u_{bb}^2 = \frac{MSamong - MSwithin}{n}$$

$MSamong$ quadratic mean of the results of homogeneity between bottle
$MSwithin$ quadratic mean of the results of homogeneity within bottle
n number of measurements per bottle

Stability

In accordance with ISO Guide 35:2017 a stability study was performed. As a result, the material was considered as stable. The uncertainty of long term stability u_{lts} was calculated.

Total expanded uncertainty

The total expanded uncertainty U_{CRM} for a confidence interval of 95% (k=2) was calculated by taking into account the uncertainty of characterization u_{char}, of inhomogeneity u_{bb} and long-term stability u_{lts} using the following formula:

$$U_{CRM} = k \times \sqrt{u_{char}^2 + u_{bb}^2 + u_{lts}^2}$$

Traceability

All of the results derived as part of this testing program have traceability to the SI unit kg/kg or BCS-CRM No. 393.

Methods used

The analytical work performed to assess this material was carried out by the FLUXANA laboratory, which works under DIN EN ISO/IEC 17025:2005 accreditation.
In accordance with DIN EN ISO 17034:2017 and ISO Guide 35:2017, we use the approach “measurement by a single (primary) method in a single laboratory”. An example for this approach is found in DIN ISO 13528:2015 E.5. Using this approach, samples of the test material that is to be the new reference material are tested along with matching and/or synthetic RMs using a suitable method. The assigned values X_{CRM} and their uncertainties U_{CRM} are then derived from a calibration against the certified reference values of the compared RMs. Synthetic RMs are made from pure chemicals by weighing.

Measurement method used: XRF fusion method for materials

This certificate is in conformance with ISO Guide 31:2015.