

FLX-2002-01-COA

DIN EN ISO 17034:2017



Certified Reference Material FLX-2002 - Limestone

Certified Values

Parameter	Mass fraction in % ¹⁾	Uncertainty in % ²⁾	Traceable to
Al ₂ O ₃	0,653	0,042	BAM Reinstoff Nr. 2
CaO	53,45	0,39	BAM Reinstoff Nr. 3
Fe ₂ O ₃	0,396	0,029	IV CGFE10 Lot R2-FE693527
K ₂ O	0,083	0,012	IV CGK1 Lot U2-K728258
MgO	0,549	0,039	BAM Reinstoff Nr. 6A
Mn ₂ O ₃	0,012	0,005	IV CGMN1 Lot U2-MN734301
P ₂ O ₅	0,011	0,004	IV CGP1 Lot U2-P735193
SiO ₂	1,74	0,07	BAM Reinstoff Nr. 1
TiO ₂	0,038	0,009	IV AAT11 Lot T2-TI720575

Table1) Certified Values

1) Certified value traceable to SI unit kg/kg based on dried material (105°C until constant mass).

2) Total expanded uncertainty U_{CRM} calculated for a confidence interval of 95% ($k=2$).

The sum of all oxides is **100,23 %**. This includes informational values and LOI.

This certificate is valid, within the uncertainty specified, **until 31.09.2034**, 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, **11.09.2024**

Responsible Reference Materials

Dr. Rainer Schramm

Quality Management

Ch. Winkels-Herding

Description of the CRM

This reference material is an industrial product and was taken directly from the production stream. The complete batch was sealed into 30 g bottles. This material is normally used as limestone.

Intended use

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

Informational Values

Parameter	Mass Fraction in % ³⁾	Uncertainty ⁴⁾
BaO	0,005 ⁵⁾	0,002 ⁵⁾
Na₂O	0,020 ⁵⁾	0,007 ⁵⁾
SO₃	0,036 ⁵⁾	0,008 ⁵⁾
SrO	0,028 ⁵⁾	0,002 ⁵⁾
LOI	43,20 ⁶⁾	

Table2) Informational Values

3) Only Informational Value.

4) Total expanded uncertainty U_{s^*} calculated for a confidence interval of 95% ($k=2$), if present.

5) Based on dried material (105°C until constant mass). Values taken from PT FXRV-2024-01

6) LOI = loss on ignition at least 18 hours at 1050°C.

Instructions for the correct use of the CRM

This material is moisture sensitive. The material has to be annealed at 1050°C for at least 18 hours to determine the LOI. When removing the material from the oven, make sure that it is placed in a desiccator suitable for limestone after a short period of remaining outside the furnace (max. 3 minutes). Please note that only special desiccants can be used, e. g. silica gel is not suitable. If no such material is available, it is better to use a desiccator without a desiccant and to process the material further as soon as it is hand warm.

The minimum sample quantity for analysis should be 0,5 g.

Recommended sample preparation with fused bead preparation and measurement with XRF

Typically, for the fused bead preparation of raw materials, the samples are annealed before use to achieve high precision. In this step, however, elements (Na₂O, K₂O and SO₃) may be lost in limestone because of the use of temperatures above 1000°C for a long time. In the case of limestone there is also a risk that moisture will be absorbed again after annealing during further processing. Therefore, we recommend an alternative method that provides a higher precision.

In this process, the limestone is fused from dried material with an additional weight of sample (corresponding to the determined LOI). The LOI takes place during the melting process and components that are otherwise lost during annealing in the muffle furnace are retained because they are bound by the flux. The sum of the components after fusion should be close to 100% if the added weight was corresponding with the determined LOI.

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 33405:2024 a homogeneity study was performed. A one-way ANOVA was used to calculate the batch inhomogeneity.

Stability

In accordance with ISO 33405:2024 a stability study was performed. As a result, the stability of the material was considered as fit for purpose. The uncertainty of long-term stability 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} .

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

Traceability

All of the certified values derived as part of this testing program have traceability to the reference materials stated in table 1.

Methods used

The analytical work performed to assess this material was carried out by the FLUXANA laboratory, which works according to ISO/IEC 17025:2018.

In accordance with ISO 17034:2017 and ISO 33405:2024, we use the approach stated in ISO 17034:2017 Chapter 7.12.3. d) value transfer from an RM to a closely matched candidate RM performed using a single measurement procedure performed by one laboratory.

An example for this approach is found in ISO 13528:2022 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 with fusion as sample preparation technique.

This certificate is in conformance with ISO 33401:2024.

feedback@fluxana.de

