

FLX-CRM 115-03

# **Certified Reference Material FLX-CRM 115 - Cement**

# **New certificate issued December 2023**

Mass fraction in %	Certified value <sup>1)</sup>	Uncertainty <sup>2)</sup>	Traceable to
Al <sub>2</sub> O <sub>3</sub>	8.26	0.08	NIST 1881A
CaO	54.79	0.23	SI unit kg/kg
Cr <sub>2</sub> O <sub>3</sub>	0.004	0.004	NIST 1881A
Fe <sub>2</sub> O <sub>3</sub>	0.987	0.145	NIST 1881A
K <sub>2</sub> O	0.622	0.088	NIST 1881A
MgO	2.98	0.13	NIST 1881A
Mn <sub>2</sub> O <sub>3</sub>	0.217	0.035	NIST 1881A
Na₂O	0.197	0.051	NIST 1881A
P <sub>2</sub> O <sub>5</sub>	0.074	0.007	NIST 1881A
SiO <sub>2</sub>	27.72	0.06	SI unit kg/kg
SrO	0.119	0.014	NIST 1881A
TiO <sub>2</sub>	0.620	0.018	NIST 1881A
ZnO	0.008	0.004	NIST 1881A

- 1) Certified value traceable to cement NIST 1881A or SI unit kg/kg based on ignited sample material (1h 950°C).
- 2) Expanded uncertainty  $U_{CRM}$  calculated for a confidence interval of 95% (k=2) based on a combined uncertainty of characterization, homogeneity and long term stability.

Bedburg-Hau, 11.12.2023

Responsible Reference Materials

Dr. Rainer Schramm

**Quality Management** Charlotte Winkels-Herding





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## **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 30g bottles. This material is normally used as cement for constructions.

#### Intended use

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

#### Instructions for the correct use of the CRM

This material is moisture sensitive. It has to be ignited for minimum 1 hour at 950°C prior to use. The ignition process must result in a constant weight. The ignited material must be stored in a desiccator not longer than 24h, then reignition might be necessary. The minimum sample quantity for analysis should be 1.0g to be in agreement with the stated uncertainties.

For XRF use, ignited samples should be prepared as a fused bead, e.g. in accordance with ISO 29581-2:2010.

The following table contains the informal values obtained directly from the original material when it was bottled:

Mass fraction in %	Info only <sup>1)</sup>	
SO₃ total	3.90	
SO <sub>4</sub> <sup>2-</sup> as SO <sub>3</sub>	2.41	
LOI	1.56	
S <sup>2-</sup>	0.611	

<sup>1)</sup> informal value based on original sample material.

### **Expiration of Certification**

This certificate is valid, within the uncertainty specified, until 31.12.2033, 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.

#### **Hazardous situation**

For this material an actual MSDS is available.





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## Level of homogeneity

In accordance with ISO Guide 35: 2006 a homogeneity study was performed. A one-way ANOVA was used to calculate the batch inhomogeneity  $u_{bb}^2$ .

$$u_{bb}^2 = \frac{MS_{among} - MS_{within}}{n}$$

 $MS_{among}$  $MS_{within}$ n quadratic mean of the results of homogeneity between bottle quadratic mean of the results of homogeneity within bottle number of measurements per bottle

## Stability

In accordance with ISO Guide 35: 2006 a stability study was performed. As a result the material was considered as stable. The uncertainty of long term stability u<sup>2</sup><sub>lts</sub> 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 from characterization  $u^2_{char}$ , from inhomogeneity  $u^2_{bb}$  and long term stability  $u^2_{lts}$  with the following formula:

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

### **Traceability**

The analytical work performed to assess this material was carried out by the FLUXANA laboratory, which works under DIN EN ISO/IEC 17025 accreditation.

All of the results derived as part of this testing program have traceability to NIST 1880B from NIST and some additionally to the SI unit kg.

#### Methods used

In accordance with ISO Guide 34, we use the approach "measurement by a single (primary) method in a single laboratory". An example for this approach is also found in DIN ISO 13528:2009-01 chapter 5.4. Using this approach, samples of the test material that is to be the new reference material are prepared first. They are tested along with CRMs 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 CRMs. The error of the calibration used can be neglected because only the differences in the results between the new reference material and the CRM or synthetic RM are part of the evaluation.

CRM used: NIST 1881A

Synthetic RMs made from pure chemicals by weighing

Measurement method used: ISO 29581-2:2010-03

This certificate is in conformance with ISO Guide 31:2000.