FLX-CRM 120-03

Certified Reference Material
FLX-CRM 120 - Cement

New certificate issued December 2023

Mass fraction in %	Certified value[1)]	Uncertainty[2)]	Traceable to
Al_2O_3	7.77	0.10	NIST 1881A
CaO	54.56	0.07	SI unit kg/kg
Cr_2O_3	0.005	0.002	NIST 1881A
Fe_2O_3	1.13	0.11	NIST 1881A
K_2O	0.704	0.032	NIST 1881A
MgO	3.62	0.09	NIST 1881A
Mn_2O_3	0.191	0.003	NIST 1881A
Na_2O	0.188	0.063	NIST 1881A
P_2O_5	0.087	0.006	NIST 1881A
SiO_2	26.97	0.08	SI unit kg/kg
SrO	0.126	0.343	NIST 1881A
TiO_2	0.609	0.004	NIST 1881A
ZnO	0.008	0.003	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
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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[1)]
SO_3 total	4.35
SO_4^{2-} as SO_3	3.18
LOI[2)]	**2.32**
S^{2-}	0.632

1) informal value based on original sample material.
2) LOI has changed since production in 2013

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.

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 $u^2{}_{bb}$.

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

MS_{among}	quadratic mean of the results of homogeneity between bottle
MS_{within}	quadratic mean of the results of homogeneity within bottle
n	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^2_{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 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.