

CERTIFICATE OF ANALYSIS

FLX-CRM 121 Cement

New certificate issued July 2019

Mass fraction in %	Certified value ¹⁾	Uncertainty ²⁾	Traceable to
Al ₂ O ₃	8.78	0.10	NIST 1881A
CaO	44.45	0.05	SI unit kg/kg
Cr ₂ O ₃	0.008	0.003	NIST 1881A
Fe ₂ O ₃	3.04	0.37	NIST 1881A
K ₂ O	2.73	0.26	NIST 1881A
MgO	1.41	0.09	NIST 1881A
Mn ₂ O ₃	0.111	0.004	NIST 1881A
Na ₂ O ³⁾	1.23	0.15	NIST 1885A
P ₂ O ₅	0.112	0.006	NIST 1881A
SiO ₂	30.81	0.10	SI unit kg/kg
SO ₃ total	2.55	0.09	NIST 1881A
SO ₄ ²⁻ as SO ₃	2.37	0.10	SI unit kg/kg
SrO	0.140	0.051	NIST 1881A
TiO ₂	0.344	0.009	NIST 1881A
ZnO	0.031	0.003	NIST 1881A
LOI ³⁾	4.24	0.21	not certified, info only
S ²⁻	0.025	0.049	not certified, info only

1) Certified value traceable to cement NIST 1881A, NIST 1885A or SI unit kg/kg based on original sample material.

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.

3) LOI is informal only and is excluded from certification because it might change over time.

The sum of all oxides is 99.97%. This includes SO₃ total and LOI and excludes SO₄²⁻ as SO₃ and S²⁻.

Bedburg-Hau, 26.07.2019

Responsible Reference Materials
Susan Aschenbrenner

General Manager
Dr. Rainer Schramm

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 **The Material has to be dried prior to use.** After opening the material has to be sealed again as soon as possible to avoid any change. Otherwise 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. **If the sample is used after ignition (950°C to constant mass), the ignited certified values have to be calculated, using the following formula:**

$$conc_{Ignited} = \frac{conc_{original} * Sum}{Sum - LOI}$$

The following table contains **an example of certified values** $conc_{original}$ recalculated to ignited sample values $conc_{Ignited}$ (**LOI=4,24%**) as obtained directly from, e.g., a fused bead.

FLX-CRM 121	Certified values for ignited sample		
mass%	conc _{Ignited}	U _{CRM}	Traceable to
Al ₂ O ₃	9.17	0.10	NIST 1881A
CaO	46.42	0.05	SI unit kg/kg
Cr ₂ O ₃	0.008	0.003	NIST 1881A
Fe ₂ O ₃	3.17	0.39	NIST 1881A
K ₂ O	2.85	0.28	NIST 1881A
MgO	1.47	0.09	NIST 1881A
Mn ₂ O ₃	0.116	0.004	NIST 1881A
Na ₂ O	1.28	0.15	NIST 1885A
P ₂ O ₅	0.117	0.007	NIST 1881A
SiO ₂	32.18	0.10	SI unit kg/kg
SO ₃ total	2.66	0.09	NIST 1881A
SO ₄ ²⁻ as SO ₃			
SrO	0.146	0.053	NIST 1881A
TiO ₂	0.360	0.009	NIST 1881A
ZnO	0.033	0.003	NIST 1881A

LOI				
S ²⁻				

Expiration of Certification

This certificate is valid, within the uncertainty specified, **until 31.05.2025**, 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}

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^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 1881A 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

Further information

The following table lists all results obtained for this sample material. Values in bold represent the results with the smallest uncertainty; i.e., those used for the certification.

Users who need all values traceable to a NIST CRM are provided with all results in the following table. However in comparison with the values traceable to the SI unit, they show a higher uncertainty.

For comparison with the certified values, the results of an independently performed proficiency test are also given. A detailed report is available at www.fluxana.com.

FLX-CRM 121	Traceable to		Traceable to		For Comparison	
	NIST 1881A		SI unit kg/kg		results of PT	
mass%	X_{CRM}	U_{CRM}	X_{CRM}	U_{CRM}	X_{PT}	U_{PT}
Al ₂ O ₃	8.78	0.10			8.78	0.11
CaO	44.55	0.35	44.45	0.05	44.76	0.29
Cr ₂ O ₃	0.008	0.003			0.009	0.002
Fe ₂ O ₃	3.04	0.37			3.08	0.04
K ₂ O	2.73	0.26			2.71	0.05
MgO	1.41	0.09			1.41	0.03
Mn ₂ O ₃	0.111	0.004			0.112	0.006
Na ₂ O	1.23³⁾	0.15			1.22	0.05
P ₂ O ₅	0.112	0.006			0.113	0.004
SiO ₂	30.96	0.18	30.81	0.10	30.74	0.24
SO ₃ total	2.55	0.09			2.53	0.05
SO ₄ ²⁻ as SO ₃	2.45 ¹⁾	0.10	2.37	0.10⁴⁾	2.49	0.08
SrO	0.140	0.051			0.137	0.007

TiO ₂	0.344	0.009			0.351	0.008
ZnO	0.031	0.003			0.03	0.003
LOI	4.34 ²⁾	0.21	4.24	0.21⁴⁾	4.15	0.06
S ²⁻					0.025	0.049
Sum (w/o SO₄²⁻+S²⁻)	100.33				100.12	

1) Traceable to NIST 1880B.

2) Traceable to NIST 1888B.

3) Traceable to NIST 1885A.

4) U_{CRM} taken from calculations based on NIST CRM.

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