

FLUXANA

CERTIFICATE OF ANALYSIS

FLX-CRM 113 Cement

Mass fraction in %	Certified value ¹⁾	Uncertainty ²⁾	Traceable to
Al ₂ O ₃	5.06	0.07	NIST 1880B
CaO	63.63	0.25	SI unit kg/kg
Cr ₂ O ₃	0.007	0.002	NIST 1880B
Fe ₂ O ₃	2.75	0.03	NIST 1880B
K ₂ O	0.619	0.025	NIST 1880B
MgO	2.49	0.06	NIST 1880B
Mn ₂ O ₃	0.233	0.020	NIST 1880B
Na ₂ O	0.092	0.027	NIST 1880B
P ₂ O ₅	0.135	0.003	NIST 1880B
SiO ₂	20.98	0.09	SI unit kg/kg
SO ₃ total	2.47	0.12	NIST 1880B
SO ₄ ²⁻ as SO ₃	2.40	0.15	SI unit kg/kg
SrO	0.064	0.019	NIST 1880B
TiO ₂	0.231	0.013	NIST 1880B
ZnO	0.030	0.002	NIST 1880B
LOI	1.53	0.08	not certified, info only
S ²⁻	0.137	0.019	not certified, info only

- 1) Certified value traceable to cement NIST 1880B 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.

The sum of all oxides is 100.31%. This includes SO₃ total and LOI and excludes SO₄²⁻ as SO₃ and S²⁻.

Bedburg-Hau, 01.06.2015

Responsible Reference Materials
Dr. Barbara Schäfer

Statistics and Report
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. 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. 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.

The following table contains the certified values $conc_{original}$ recalculated to ignited sample values $conc_{ignited}$ as obtained directly from, e.g., a fused bead by the following formula:

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

FLX-CRM 113	Certified values for ignited sample			
	mass%	conc _{ignited}	U _{CRM}	Traceable to
Al ₂ O ₃	5.14	0.07		NIST 1880B
CaO	64.61	0.25		SI unit kg/kg
Cr ₂ O ₃	0.007	0.002		NIST 1880B
Fe ₂ O ₃	2.80	0.03		NIST 1880B
K ₂ O	0.628	0.026		NIST 1880B
MgO	2.52	0.06		NIST 1880B
Mn ₂ O ₃	0.237	0.021		NIST 1880B
Na ₂ O	0.094	0.027		NIST 1880B
P ₂ O ₅	0.137	0.003		NIST 1880B
SiO ₂	21.31	0.09		SI unit kg/kg
SO ₃ total	2.51	0.13		NIST 1880B
SO ₄ ²⁻ as SO ₃				
SrO	0.065	0.019		NIST 1880B
TiO ₂	0.234	0.013		NIST 1880B
ZnO	0.030	0.002		NIST 1880B
LOI				
S ²⁻				
Sum	100.31			

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}

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

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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 1880B**

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 113	Traceable to		Traceable to		For Comparison	
	NIST 1880B		SI unit kg/kg		results of PT	
mass%	X_{CRM}	U_{CRM}	X_{CRM}	U_{CRM}	X_{PT}	U_{PT}
Al ₂ O ₃	5.06	0.07			5.04	0.05
CaO	63.62	0.47	63.63	0.25	63.68	0.27
Cr ₂ O ₃	0.007	0.002			0.008	0.001
Fe ₂ O ₃	2.76	0.03			2.76	0.03
K ₂ O	0.619	0.025			0.586	0.028
MgO	2.49	0.06			2.45	0.03
Mn ₂ O ₃	0.233	0.020			0.22	0.02
Na ₂ O	0.092	0.027			0.106	0.031
P ₂ O ₅	0.135	0.003			0.137	0.007
SiO ₂	20.99	0.37	20.98	0.09	21.10	0.14
SO ₃ total	2.47	0.12			2.55	0.07
SO ₄ ²⁻ as SO ₃	2.40	0.15	2.40	0.15¹⁾		
SrO	0.064	0.019			0.063	0.01
TiO ₂	0.231	0.013			0.231	0.01
ZnO	0.030	0.002			0.028	0.004
LOI	1.58	0.08	1.53	0.08¹⁾	1.47	0.06
S ²⁻					0.137	0.019
Sum (w/o SO ₄ ²⁻ +S ²⁻)	100.36				100.43	

1) U_{CRM} taken from the calculations based on NIST 1880B.

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