

FLUXANA

CERTIFICATE OF ANALYSIS FLX-CRM 130 Cement

Mass fraction in %	Certified value ¹⁾	Uncertainty ²⁾	Traceable to
Al ₂ O ₃	11.62	0.11	NIST 1881A
CaO	56.60	0.27	SI unit kg/kg
Cr ₂ O ₃	0.021	0.020	NIST 1881A
Fe ₂ O ₃	2.88	0.12	NIST 1881A
K ₂ O	0.682	0.038	NIST 1881A
MgO	1.84	0.08	NIST 1881A
Mn ₂ O ₃	0.062	0.002	NIST 1881A
Na ₂ O	0.277	0.096	NIST 1881A
P ₂ O ₅	0.067	0.006	NIST 1881A
SiO ₂	14.35	0.15	SI unit kg/kg
SO ₃ total	10.91	0.27	NIST 1881A
SO ₄ ²⁻ as SO ₃	10.18 ³⁾	0.40	not certified, info only
SrO	0.052	0.006	NIST 1881A
TiO ₂	0.563	0.005	NIST 1881A
ZnO	0.018	0.003	NIST 1881A
LOI	5.12	0.29	not certified, info only

- 1) Certified value traceable to cement NIST 1881A or SI unit kg/kg based on ignited sample material for 1 hour at 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.
- 3) Value traceable to SI unit kg/kg based on original sample material.

The sum of all oxides is 99.94%. This includes SO₃ total and excludes SO₄²⁻ as SO₃ and LOI.

Bedburg-Hau, 28.04.2016

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. This material has to be ignited for minimum 1 hour at 950°C prior use. The ignition process must result in a constant weight. The ignited material must be stored in a desiccator not longer than 24h, then re-ignition might be necessary. The minimum sample quantity for analysis should be 0.5g.

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

Expiration of Certification

This certificate is valid, within the uncertainty specified, **until 28.04.2026**, 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_{ITS} 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 130	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	11.62	0.11			11.71	0.19
CaO	56.48	0.43	56.60	0.27	56.75	0.47
Cr ₂ O ₃	0.021	0.020			0.020	0.004
Fe ₂ O ₃	2.88	0.12			2.89	0.09
K ₂ O	0.682	0.038			0.701	0.014
MgO	1.84	0.08			1.85	0.06
Mn ₂ O ₃	0.062	0.002			0.065	0.006
Na ₂ O	0.277	0.096			0.285	0.041
P ₂ O ₅	0.067	0.006			0.072	0.014
SiO ₂	14.36	0.22	14.35	0.15	14.39	0.25
SO ₃ total	10.91	0.27			11.03	0.31
SO ₄ ²⁻ as SO ₃			10.18	0.40		
SrO	0.052	0.006			0.052	0.004
TiO ₂	0.563	0.005			0.565	0.010
ZnO	0.018	0.003			0.018	0.004
LOI			5.12	0.29	5.22	0.16
Sum (w/o SO ₄ ²⁻)	99.83				100.40	

This certificate is in conformance with ISO Guide 31:2015.

FLUXANA

CERTIFICATE OF ANALYSIS FLX-CRM 131 Cement

Mass fraction in %	Certified value ¹⁾	Uncertainty ²⁾	Traceable to
Al ₂ O ₃	23.10	0.21	NIST 1881A
CaO	42.89	0.09	SI unit kg/kg
Cr ₂ O ₃	0.038	0.020	NIST 1881A
Fe ₂ O ₃	3.24	0.11	NIST 1881A
K ₂ O	0.287	0.054	NIST 1881A
MgO	1.62	0.08	NIST 1881A
Mn ₂ O ₃	0.029	0.002	NIST 1881A
Na ₂ O	0.466	0.080	NIST 1881A
P ₂ O ₅	0.060	0.006	NIST 1881A
SiO ₂	8.73	0.13	SI unit kg/kg
SO ₃ total	18.19	0.43	NIST 1881A
SO ₄ ²⁻ as SO ₃	17.85 ³⁾	0.73	not certified, info only
SrO	0.067	0.005	NIST 1881A
TiO ₂	1.15	0.01	NIST 1881A
ZnO	0.006	0.003	NIST 1881A
LOI	2.03	0.16	not certified, info only

1) Certified value traceable to cement NIST 1881A or SI unit kg/kg based on ignited sample material for 1 hour at 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.

3) Value traceable to SI unit kg/kg based on original sample material.

The sum of all oxides is 99.87%. This includes SO₃ total and excludes SO₄²⁻ as SO₃ and LOI.

Bedburg-Hau, 28.04.2016

Responsible Reference Materials

Dr. Barbara Schäfer



Statistics and Report

Dr. Rainer Schramm



Description of the CRM

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Instructions for the correct use of the CRM

This material is moisture sensitive. This material has to be ignited for minimum 1 hour at 950°C prior use. The ignition process must result in a constant weight. The ignited material must be stored in a desiccator not longer than 24h, then re-ignition might be necessary. The minimum sample quantity for analysis should be 0.5g.

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

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

Traceability

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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.

FLUXANA® GmbH & Co. KG, Borschelstr. 3, 47551 Bedburg-Hau, Germany

Your supplier for XRF Application Solutions

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 131	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	23.10	0.21			23.16	0.47
CaO	42.67	0.35	42.89	0.09	43.07	0.69
Cr ₂ O ₃	0.038	0.020			0.037	0.009
Fe ₂ O ₃	3.24	0.11			3.31	0.14
K ₂ O	0.287	0.054			0.311	0.013
MgO	1.62	0.08			1.68	0.05
Mn ₂ O ₃	0.029	0.002			0.035	0.008
Na ₂ O	0.466	0.080			0.467	0.047
P ₂ O ₅	0.060	0.006			0.064	0.009
SiO ₂	8.74	0.20	8.73	0.13	8.66	0.31
SO ₃ total	18.19	0.43			18.46	0.47
SO ₄ ²⁻ as SO ₃			17.85	0.73		
SrO	0.067	0.005			0.072	0.012
TiO ₂	1.15	0.01			1.18	0.04
ZnO	0.006	0.003			0.007	0.003
LOI			2.03	0.21	2.10	0.10
Sum (w/o SO₄²⁻)	99.67				100.52	

This certificate is in conformance with ISO Guide 31:2015.