

# FLUXANA

## CERTIFICATE OF ANALYSIS

### FLX-CRM 116 Cement

Mass fraction in %	Certified value <sup>1)</sup>	Uncertainty <sup>2)</sup>	Traceable to
Al <sub>2</sub> O <sub>3</sub>	9.11	0.13	NIST 1881A
CaO	50.05	0.08	SI unit kg/kg
Cr <sub>2</sub> O <sub>3</sub>	0.005	0.002	NIST 1881A
Fe <sub>2</sub> O <sub>3</sub>	0.86	0.12	NIST 1881A
K <sub>2</sub> O	0.693	0.129	NIST 1881A
MgO	4.47	0.20	NIST 1881A
Mn <sub>2</sub> O <sub>3</sub>	0.238	0.035	NIST 1881A
Na <sub>2</sub> O	0.201	0.049	NIST 1881A
P <sub>2</sub> O <sub>5</sub>	0.034	0.007	NIST 1881A
SiO <sub>2</sub>	30.81	0.20	SI unit kg/kg
SO <sub>3</sub> total	3.14	0.24	not certified, info only
SO <sub>4</sub> <sup>2-</sup> as SO <sub>3</sub>	1.22	0.02	SI unit kg/kg
SrO	0.091	0.036	NIST 1881A
TiO <sub>2</sub>	0.690	0.011	NIST 1881A
ZnO	0.015	0.004	NIST 1881A
LOI	-0.394	0.315	not certified, info only
S <sup>2-</sup>	0.737	0.249	not certified, info only

1) Certified value traceable to cement NIST 1881A 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 100.02%. This includes SO<sub>3</sub> total and LOI and excludes SO<sub>4</sub><sup>2-</sup> as SO<sub>3</sub> and S<sup>2-</sup>.

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 116	Certified values for ignited sample			
	mass%	$conc_{ignited}$	$U_{CRM}$	Traceable to
<b>Al<sub>2</sub>O<sub>3</sub></b>	9.08	0.13		NIST 1881A
<b>CaO</b>	49.85	0.08		SI unit kg/kg
<b>Cr<sub>2</sub>O<sub>3</sub></b>	0.005	0.002		NIST 1881A
<b>Fe<sub>2</sub>O<sub>3</sub></b>	0.860	0.123		NIST 1881A
<b>K<sub>2</sub>O</b>	0.690	0.129		NIST 1881A
<b>MgO</b>	4.45	0.20		NIST 1881A
<b>Mn<sub>2</sub>O<sub>3</sub></b>	0.237	0.035		NIST 1881A
<b>Na<sub>2</sub>O</b>	0.200	0.049		NIST 1881A
<b>P<sub>2</sub>O<sub>5</sub></b>	0.034	0.007		NIST 1881A
<b>SiO<sub>2</sub></b>	30.69	0.20		SI unit kg/kg
<b>SO<sub>3</sub> total</b>				
<b>SO<sub>4</sub><sup>2-</sup> as SO<sub>3</sub></b>				
<b>SrO</b>	0.091	0.036		NIST 1881A
<b>TiO<sub>2</sub></b>	0.687	0.011		NIST 1881A
<b>ZnO</b>	0.015	0.004		NIST 1881A
<b>LOI</b>				
<b>S<sup>2-</sup></b>				

## **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 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](http://www.fluxana.com).

FLX-CRM 116	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 <sub>2</sub> O <sub>3</sub>	<b>9.11</b>	<b>0.13</b>			9.06	0.10
CaO	50.05	0.35	<b>50.05</b>	<b>0.08</b>	49.86	0.32
Cr <sub>2</sub> O <sub>3</sub>	<b>0.005</b>	<b>0.002</b>			0.007	0.003
Fe <sub>2</sub> O <sub>3</sub>	<b>0.864</b>	<b>0.123</b>			0.966	0.023
K <sub>2</sub> O	<b>0.693</b>	<b>0.129</b>			0.662	0.028
MgO	<b>4.47</b>	<b>0.20</b>			4.40	0.05
Mn <sub>2</sub> O <sub>3</sub>	<b>0.238</b>	<b>0.035</b>			0.221	0.023
Na <sub>2</sub> O	<b>0.201</b>	<b>0.049</b>			0.208	0.036
P <sub>2</sub> O <sub>5</sub>	<b>0.034</b>	<b>0.007</b>			0.029	0.004
SiO <sub>2</sub>	30.88	0.25	<b>30.81</b>	<b>0.20</b>	30.71	0.18
SO <sub>3</sub> total					<b>3.14</b>	<b>0.24</b>
SO <sub>4</sub> <sup>2-</sup> as SO <sub>3</sub>	1.30 <sup>1)</sup>	0.020	<b>1.22</b>	<b>0.02<sup>3)</sup></b>		
SrO	<b>0.091</b>	<b>0.036</b>			0.081	0.012
TiO <sub>2</sub>	<b>0.690</b>	<b>0.011</b>			0.683	0.018
ZnO	<b>0.015</b>	<b>0.004</b>			0.016	0.003
LOI	-0.337 <sup>2)</sup>	0.455	<b>-0.394</b>	<b>0.315<sup>3)</sup></b>	-0.164	0.175
S <sup>2-</sup>					<b>0.737</b>	<b>0.249</b>
Sum (w/o SO <sub>4</sub> <sup>2-</sup> +S <sup>2-</sup> )					99.89	

1) Traceable to NIST 1880B.      2) Traceable to NIST 1888B.

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

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