

# CERTIFICATE OF ANALYSIS

## FLX-137

### Certified Values

	Mass fraction in % <sup>1)</sup>	Uncertainty <sup>2)</sup>	Traceable to
Al <sub>2</sub> O <sub>3</sub>	4,99	0,07	SI Unit kg/kg
CaO	64,77	0,23	SI Unit kg/kg
Fe <sub>2</sub> O <sub>3</sub>	3,07	0,16	NIST1885b
K <sub>2</sub> O	0,769	0,024	NIST1885b
MgO	1,64	0,02	SI Unit kg/kg
Mn <sub>2</sub> O <sub>3</sub>	0,266	0,017	NIST1885b
P <sub>2</sub> O <sub>5</sub>	0,171	0,007	NIST1885b
SiO <sub>2</sub>	20,78	0,10	SI Unit kg/kg
SO <sub>3</sub>	3,17	0,10	NIST1885b
SrO	0,076	0,026	NIST1885b
TiO <sub>2</sub>	0,221	0,010	NIST1885b
ZnO	0,029	0,003	NIST1885b

- 1) Certified value traceable to Cement NIST1885b and SI unit kg/kg based on ignited sample material for 1h at 950°C.
- 2) Expanded uncertainty  $U_{CRM}$  calculated for a confidence interval of 95% (k=2) based on uncertainty of characterization.

The sum of all oxides is 100,06%. This also includes informational values.

This certificate is valid, within the uncertainty specified, **until 02.05.2028**, 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.

Bedburg-Hau, 02.05.2018

**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.

### Informational Values

	Mass Fraction in % <sup>3)</sup>	Uncertainty <sup>4)</sup>
Cr <sub>2</sub> O <sub>3</sub>	0,007	0,002
Na <sub>2</sub> O	0,107	0,067
LOI	2,66	-

3) Only Informational Value.

4) Expanded uncertainty  $U_{CRM}$  calculated for a confidence interval of 95% (k=2) based on uncertainty of characterization, if present.

## 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 reignition 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.

## 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  $u_{bb}^2$ .

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

$MS_{among}$  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  $u_{char}^2$ , from inhomogeneity  $u_{bb}^2$  and long-term stability  $u_{lts}^2$  with the following formula:

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

### 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 the SI unit kg.

### Methods used

In accordance with ISO 17034 and ISO Guide 35, 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 matching and/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 compared RMs. The error of the calibration used can be neglected because only the differences in the results between the new reference material and the matching/synthetic RM are part of the evaluation. Synthetic RMs are made from pure chemicals by weighing.

Measurement method used: XRF fusion method for materials

### Further information

The following table lists all results obtained for this sample material. Values in **bold** represent the results used for the certification. Results in *italic* are informational values.

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 mostly 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-137	Tracable to NIST 1885B		Tracable to SI unit kg/kg		For Comparison: results of PT	
	$X_{CRM}$	$U_{CRM}$	$X_{CRM}$	$U_{CRM}$	$X_{PT}$	$U_{PT}$
mass%						
Al <sub>2</sub> O <sub>3</sub>	4,96	0,17	4,99	0,07	5,02	0,05
CaO	64,96	0,24	64,77	0,23	64,67	0,22
Cr <sub>2</sub> O <sub>3</sub>	0,007	0,002			0,008	0,001
Fe <sub>2</sub> O <sub>3</sub>	3,07	0,16			3,05	0,05
K <sub>2</sub> O	0,769	0,024			0,782	0,042
MgO	1,57	0,11	1,64	0,02	1,61	0,03
Mn <sub>2</sub> O <sub>3</sub>	0,266	0,017			0,257	0,007
Na <sub>2</sub> O	0,093	0,072	0,107	0,067	0,091	0,036
P <sub>2</sub> O <sub>5</sub>	0,171	0,007			0,172	0,002
SiO <sub>2</sub>	20,54	0,41	20,78	0,10	20,61	0,12
SO <sub>3</sub>	3,17	0,10			3,22	0,06
SrO	0,076	0,026			0,078	0,003
TiO <sub>2</sub>	0,221	0,010			0,225	0,004
ZnO	0,029	0,003			0,030	0,002
LOI	2,66	-			2,51	
Summe	99,89		-		99,82	

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