

## CERTIFICATE OF ANALYSIS

### FLX-CRM 130 Cement

New certificate issued January 2021

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

Bedburg-Hau, 13.01.2021

#### Responsible Reference Materials

Susan Aschenbrenner

#### 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 50 ml 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 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.

## 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  $u^2_{bb}$ .

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

$MS_{among}$

$MS_{within}$

$n$

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

| FLX-CRM 130                                      | Traceable to     |                  | Traceable to     |                  | For Comparison  |                 |
|--------------------------------------------------|------------------|------------------|------------------|------------------|-----------------|-----------------|
|                                                  | NIST 1881A       |                  | SI unit kg/kg    |                  | results of PT   |                 |
| mass%                                            | X <sub>CRM</sub> | U <sub>CRM</sub> | X <sub>CRM</sub> | U <sub>CRM</sub> | X <sub>PT</sub> | U <sub>PT</sub> |
| Al <sub>2</sub> O <sub>3</sub>                   | <b>11.62</b>     | <b>0.11</b>      |                  |                  | 11.71           | 0.19            |
| CaO                                              | 56.48            | 0.43             | <b>56.60</b>     | <b>0.27</b>      | 56.75           | 0.47            |
| Cr <sub>2</sub> O <sub>3</sub>                   | <b>0.021</b>     | <b>0.020</b>     |                  |                  | 0.020           | 0.004           |
| Fe <sub>2</sub> O <sub>3</sub>                   | <b>2.88</b>      | <b>0.12</b>      |                  |                  | 2.89            | 0.09            |
| K <sub>2</sub> O                                 | <b>0.682</b>     | <b>0.038</b>     |                  |                  | 0.701           | 0.014           |
| MgO                                              | <b>1.84</b>      | <b>0.08</b>      |                  |                  | 1.85            | 0.06            |
| Mn <sub>2</sub> O <sub>3</sub>                   | <b>0.062</b>     | <b>0.002</b>     |                  |                  | 0.065           | 0.006           |
| Na <sub>2</sub> O                                | <b>0.277</b>     | <b>0.096</b>     |                  |                  | 0.285           | 0.041           |
| P <sub>2</sub> O <sub>5</sub>                    | <b>0.067</b>     | <b>0.006</b>     |                  |                  | 0.072           | 0.014           |
| SiO <sub>2</sub>                                 | 14.36            | 0.22             | <b>14.35</b>     | <b>0.15</b>      | 14.39           | 0.25            |
| SO <sub>3</sub> total                            | <b>10.91</b>     | <b>0.27</b>      |                  |                  | 11.03           | 0.31            |
| SO <sub>4</sub> <sup>2-</sup> as SO <sub>3</sub> |                  |                  | <b>10.18</b>     | <b>0.40</b>      |                 |                 |
| SrO                                              | <b>0.052</b>     | <b>0.006</b>     |                  |                  | 0.052           | 0.004           |
| TiO <sub>2</sub>                                 | <b>0.563</b>     | <b>0.005</b>     |                  |                  | 0.565           | 0.010           |
| ZnO                                              | <b>0.018</b>     | <b>0.003</b>     |                  |                  | 0.018           | 0.004           |
| LOI                                              |                  |                  | <b>5.12</b>      | <b>0.29</b>      | 5.22            | 0.16            |
| <b>Sum (w/o SO<sub>4</sub><sup>2-</sup>)</b>     | 99.83            |                  |                  |                  | 100.40          |                 |

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