

**CERTIFICATE OF ANALYSIS**  
**FLX-CRM 100**  
Cement

**Certified Values**

	Mass fraction in mass% <sup>1)</sup>	Uncertainty in mass% <sup>2)</sup>
<b>Al<sub>2</sub>O<sub>3</sub></b>	5,54	0,03
<b>CaO</b>	64,51	0,14
<b>Cr<sub>2</sub>O<sub>3</sub></b>	0,009	0,002
<b>Fe<sub>2</sub>O<sub>3</sub></b>	2,62	0,01
<b>K<sub>2</sub>O</b>	0,82	0,11
<b>MgO</b>	1,47	0,02
<b>Mn<sub>2</sub>O<sub>3</sub></b>	0,066	0,004
<b>Na<sub>2</sub>O</b>	0,23	0,02
<b>P<sub>2</sub>O<sub>5</sub></b>	0,166	0,006
<b>SO<sub>3</sub></b>	2,97	0,03
<b>SiO<sub>2</sub></b>	20,89	0,06
<b>SrO</b>	0,286	0,003
<b>TiO<sub>2</sub></b>	0,283	0,004
<b>ZnO</b>	0,051	0,002

- 1) Certified value based on ignited sample (1h 950°C). The values are the best estimates of the true content for each element. Each value is a panel consensus, based on the averaged results of an interlaboratory testing programme.
- 2) Uncertainty  $u_{CRM}$  calculated for a confidence interval of 95% ( $k=1$ ) based on uncertainty of characterization.

The sum of all oxides is **99,91%**.

This certificate is valid, within the uncertainty specified, **until March 2029**, 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, **04.03.2019**



**Responsible Reference Materials**  
Susan Aschenbrenner



**General Manager**  
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 50g 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>
<b>Cl</b>	0,09
<b>LOI</b>	2,37

3) Only Informational Value.

### 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.

### Storage Information

The material has to be stored in a dry and clean environment.

### Hazardous situation

For this material an actual MSDS is available.

### **Level of homogeneity**

Approximately 5% of all bottles were selected for homogeneity testing.

The batch was checked for uniformity using a wavelength -dispersive XRF unit and a test method in conformance with DIN EN ISO 29581-2:2007.

Using the data from each sample, standard deviation values were derived for each element as an indicator of any non-homogeneity (as determined for the specific sample size taken by the spectrometer).

### **Estimation of Uncertainties**

Each element certified had been analyzed by several laboratories and 95% half-width confidence intervals ( $C_{95\%}$ ) for the resultant mean values have been derived by the method shown on page 4.

The final uncertainty for each element has been derived by combining the degree of non-homogeneity and the uncertainty of the characterization, using a square-root of summed squares.

### **Traceability**

The analytical work performed to assess this material has been carried out by competent laboratories, both from the cement industry and the independent sectors. All of the results derived as part of this testing program have traceability to NIST and other national standards, as part of the analytical calibration or process control.

### **Methods used**

XRF analysis was performed by a panel of competent laboratories using the fused bead method after ignition at 950°C for 1 hour, in accordance with DIN EN ISO 29581-1: 2007. In all cases, measurement was done by WD-XRF. All XRF units were calibrated using NIST, BCS and JCA CRMs prepared by the same method. The measuring conditions were optimized to achieve the lowest measurement error possible. One sample was analyzed after dissolution, using ICP-AES and other “wet” methods as described on page 4. The results were adjusted to allow for ignition loss and thereby ensure parity with the values derived by XRF.

This Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2000 and ISO Guide 35-1989, considering the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

This certification is applicable to the whole of the sample.

Pre-ignition, this sample contains 0.06% of its sulfur as sulfide ( $S^{2-}$ ), as determined by a single test using the volumetric method with iodine.

Analytical Data

Sample	Al <sub>2</sub> O <sub>3</sub>	CaO	Cr <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Mn <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O
1	5,45*	64,25	0,005	5,57	0,67	1,44	0,060	0,20
2	5,51	64,29	0,006	2,61	0,70	1,45	0,063	0,21
3	5,52	64,36	0,008	2,61	0,75	1,46	0,064	0,22*
4	5,54	64,43	0,009	2,61	0,75	1,46	0,064	0,23
5	5,54	64,43	0,010	2,61	0,77*	1,46*	0,064	0,23
6	5,54	64,43	0,010	2,61*	0,81	1,47	0,066	0,23
7	5,54	64,45	0,010	2,62	0,84	1,47	0,067	0,23
8	5,55	64,53	0,010*	2,62	0,84	1,47	0,070	0,24
9	5,55	64,57		2,62	0,86	1,48	0,080*	0,25
10	5,55	64,58		2,62	0,87	1,48		0,25
11	5,56	64,60		2,63	0,89	1,49		0,27
12	5,57	64,60		2,64	0,89	1,49		
13	5,58	64,66		2,65	0,89	1,50		
14	5,61	64,90*		2,65	0,90	1,52		
Mean	5,54	64,51	0,009	2,62	0,82	1,47	0,066	0,23
Std Dev	0,04	0,17	0,002	0,02	0,08	0,02	0,006	0,02
C(95%)	0,02	0,10	0,002	0,01	0,04	0,01	0,004	0,01

Sample	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	SiO <sub>2</sub>	SrO	TiO <sub>2</sub>	ZnO	Cl	LOI
1	0,759	2,86*	20,74*	0,280	0,272	0,048	0,09*	2,21
2	0,160	2,88	20,78	0,282	0,278	0,050		2,24
3	0,160	2,93	20,82	0,282	0,279	0,051		2,26
4	0,160	2,95	20,85	0,285	0,280	0,052		2,34
5	0,162	2,97	20,87	0,288	0,283	0,052		2,44
6	0,162	2,97	20,89	0,290	0,285	0,053		2,49
7	0,163	2,98	20,92	0,290	0,286	0,053		2,63
8	0,170	2,99	20,93	0,290*	0,290			
9	0,177	2,99	20,93		0,290			
10	0,185*	3,00	20,93		0,290*			
11		3,00	20,94					
12		3,02	20,95					
13		3,03	20,98					
14		3,06	20,98					
Mean	0,166	2,97	20,89	0,286	0,283	0,051	0,09	2,37
Std Dev	0,009	0,05	0,07	0,004	0,006	0,002		0,15
C(95%)	0,006	0,03	0,04	0,003	0,004	0,002		0,14

Note: C(95%) is the 95% half-width confidence interval derived from the equation  $C(95\%) = (t \times SD) / \sqrt{n}$ , where n is the number of available values, t is the Student's t value for n-1 degrees of freedom and SD is the standard deviation of the tests results .

Results were derived by fused bead XRF to DIN EN ISO 29581-2: 2007.

Results marked \* were derived by methods involving dissolution, as follows:

Na and K were analysed by flame photometry; Sulfate (SO<sub>3</sub>) was analyzed gravimetrically as BaSO<sub>4</sub>; Chloride (Cl) was analyzed volumetrically using thiocyanate after Ag precipitation. Other elements were analysed by ICP-AES.

The packaging, analysis and storage of this product were supervised by R Schramm, PhD, Director. Fluxana GmbH & Co. KG, Kleve, Germany.

The certification of this product was performed by C Eveleigh PhD, Technical Director, MBH Analytical Ltd, Barnet, UK.

The monitoring of this product was done by Fluxana GmbH & Co. KG, Bedburg-Hau, Germany.

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