FLUXANA® GmbH & Co. KG, Borschelstr. 3, D-47551 Bedburg-Hau, Germany
Your supplier for XRF Application Solutions

FLUXANA

CERTIFICATE OF ANALYSIS

FLX-CRM 122 Cement

Mass fraction in %	Certified value[1)]	Uncertainty[2)]	Traceable to
Al_2O_3	5.60	0.09	NIST 1881A
CaO	59.00	0.26	SI unit kg/kg
Cr_2O_3	0.004	0.003	NIST 1881A
Fe_2O_3	1.67	0.12	NIST 1881A
K_2O	0.900	0.030	NIST 1881A
MgO	2.02	0.08	NIST 1881A
Mn_2O_3	0.111	0.006	NIST 1881A
Na_2O	0.204	0.042	NIST 1881A
P_2O_5	0.066	0.009	NIST 1881A
SiO_2	21.94	0.09	SI unit kg/kg
SO_3 total	*3.27*	*0.07*	not certified, info only
SO_4^{2-} as SO_3	2.62	0.28	SI unit kg/kg
SrO	0.131	0.113	NIST 1881A
TiO_2	0.353	0.014	NIST 1881A
ZnO	0.027	0.004	NIST 1881A
LOI	*4.86*	*0.32*	not certified, info only
S^{2-}	*0.288*	*0.087*	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 100.15%. This includes SO_3 total and LOI and excludes SO_4^{2-} as SO_3 and S^{2-}.

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 122	**Certified values for ignited sample**		
mass%	$conc_{ignited}$	U_{CRM}	Traceable to
Al_2O_3	5.88	0.09	NIST 1881A
CaO	62.01	0.27	SI unit kg/kg
Cr_2O_3	0.005	0.003	NIST 1881A
Fe_2O_3	1.75	0.12	NIST 1881A
K_2O	0.946	0.031	NIST 1881A
MgO	2.12	0.09	NIST 1881A
Mn_2O_3	0.116	0.006	NIST 1881A
Na_2O	0.214	0.044	NIST 1881A
P_2O_5	0.069	0.009	NIST 1881A
SiO_2	23.06	0.09	SI unit kg/kg
SO_3 total			
SO_4^{2-} as SO_3			
SrO	0.137	0.119	NIST 1881A
TiO_2	0.371	0.014	NIST 1881A
ZnO	0.028	0.004	NIST 1881A
LOI			
S^{2-}			

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.

FLX-CRM 122	**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_2O_3	**5.60**	**0.09**			5.63	0.08
CaO	58.96	0.43	**59.00**	**0.26**	59.17	0.21
Cr_2O_3	**0.004**	**0.003**			0.007	0.002
Fe_2O_3	**1.67**	**0.12**			1.76	0.02
K_2O	**0.900**	**0.030**			0.867	0.027
MgO	**2.02**	**0.08**			2.00	0.03
Mn_2O_3	**0.111**	**0.006**			0.112	0.004
Na_2O	**0.204**	**0.042**			0.189	0.027
P_2O_5	**0.066**	**0.009**			0.067	0.004
SiO_2	21.92	0.17	**21.94**	**0.09**	21.91	0.15
SO_3 total					**3.27**	**0.07**
SO_4^{2-} as SO_3	2.70[1)]	0.28	**2.62**	**0.28[3)]**	2.68	0.11
SrO	**0.131**	**0.113**			0.126	0.007
TiO_2	**0.353**	**0.014**			0.356	0.008
ZnO	**0.027**	**0.004**			0.027	0.003
LOI	4.97[2)]	0.32	**4.86**	**0.32[3)]**	4.65	0.06
S^{2-}					**0.288**	**0.087**
Sum (w/o SO_4^{2-}+S^{2-})					100.13	

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.