Certificate of Analysis

FLX-CRM 103

New certificate issued February 2022

Reference Material Information

Type:	Cement
Form and Size:	Granulate, as-produced, 50g
Manufactured by:	Lafarge Ciment, La Malle, France
Packaged and tested by:	FLUXANA GmbH & Co.KG, Germany
Certified by (2009):	MBH Analytical Limited, UK
Recertified by (2012):	FLUXANA GmbH & Co.KG, Germany

Certified values and their uncertainties

Percentage element by weight

Constituent	Al2O3	CaO	Fe2O3	K2O	Na2O	SiO2	SO3
Value[1]	**7.75**	**54.90**	**1.78**	**0.77**	**0.33**	**26.95**	**2.73**
Uncertainty[2]	0.06	0.16	0.04	0.07	0.02	0.11	0.07

Constituent	MgO	SrO	TiO2	Cr2O3	Mn2O3	P2O5	ZnO
Value[1]	**4.44**	**0.070**	**0.372**	**0.007**	**0.170**	**0.09**	**0.014**
Uncertainty[2]	0.04	0.001	0.005	0.002	0.006	0.01	0.003

Notes: all values are recalculated on base of ignited sample.

Definitions

1 The above values are the present best estimates of the true content for each component. Each value is a panel consensus, based on the averaged results of an inter laboratory testing program, detailed in values obtained by individual laboratories or methods.

2 The uncertainty values are coming from the half width confidence interval C(95%). It is equal to C(95%) = (t x s)/√n where t is the appropriate Student's value, n the number of acceptable mean values and s the standard deviation.

Fluxana GmbH & Co.KG on 12th February 2012

Dr. Rainer Schramm

Reissued: Fluxana GmbH & Co.KG Susan Aschenbrenner on 25th February 2022

Method of Preparation

This reference material sample was produced from commercial product. Material was taken directly from the production stream, and the complete batch was sealed into 50g bottles.

Sampling

Approximately 5% of all bottles were selected for homogeneity testing. Further samples were submitted to several laboratories for compositional analysis.

Homogeneity

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).

Chemical Analysis

XRF analysis was performed by a panel of competent laboratories using the fused bead method after ignition at 950C for 1 hour, in accordance with DIN EN ISO 29581-2: 2007. In all cases, measurement was 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 analysed 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.

The individual values listed overpage are the average of each analyst's results.

Estimation of Uncertainties

Each element certified has been analysed 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 3.

As a separate exercise, the degree of non-homogeneity of the batch for each element has been quantified by a

programme of application testing, described above.

The final uncertainty for each element has been derived by combining these two factors, using the square-root of the 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 programme have traceability to NIST and other national standards, as part of the analytical calibration or process control.

Usage

Intended use: With X-ray fluorescence spectrometers, or with methods involving dissolution.

For XRF use, samples should be ignited at 950C for 1 hour, prior to testing. Samples should be prepared as a fused bead, using 1 part sample + 8 parts Lithium tetraborate, prepared on an automated fusion machine, and otherwise in accordance with ISO 29581-2: 2007. Samples may alternatively be prepared by manual fusion in a muffle furnace at a temperature not exceeding 1100C; but with this method there is a probability that results will show higher error.

Fused beads may be stored in accordance with ISO 29581-2.

Analytical Data

Percentage element by weight

Sample	Al_2O_3	CaO	Fe_2O_3	K_2O	Na_2O	SiO_2	SO_3	S2-	MgO
1	7.49[2]	54.49	1.61	0.70	0.31	26.52[2]	2.63	0,30[2]	4.32[2]
2	7.63	54.54	1.68[2]	0.72[2]	0.31	26.58[2]	2.66	0,31[2]	4.35
3	7.66	54.55	1.74	0.72[2]	0.32	26.65[2]	2.67[4]	0,32[2]	4.36
4	7.66	54.56	1.75	0.73[2]	0.32	26.77[1]	2.70	0,34[2]	4.38
5	7.68	54.56	1.76	0.75	0.32[1]	26.83	2.72	0,38[2]	4.38
6	7.70	54.62[2]	1.77	0.76[1]	0.33	26.86	2.72[4]		4.40[2]
7	7.71	54.69	1.77	0.77[2]	0.33	26.87	2.74		4.42
8	7.72	54.69	1.77[1]	0.77	0.33[2]	26.88	2.76		4.42
9	7.73[3]	54.80	1.77	0.77	0.34[2]	26.90	2.76		4.43
10	7.74	54.82	1.77	0.78	0.34	26.91	2.77[4]		4.44[2]
11	7.78	54.87[2]	1.77	0.79	0.34	26.94	2.80[4]		4.46
12	7.79	55.00	1.78	0.81	0.35[2]	26.94			4.49
13	7.85[2]	55.08	1.80[2]	0.82[2]		26.97			4.55
14	7.88[1]		1.81	0.83		27.00			
15			1.89[1]	0.86[3]		27.04			
16			1.90[2]			27.15			
Mean	**7.72**	**54.71**	**1.77**	**0.77**	**0.33**	**26.86**			**4.42**
Std Dev	0.10	0.19	0.07	0.04	0.01	0.16			0.06
C (95%)	0.06	0.11	0.04	0.02	0.01	0.09			0.04

Sample	SrO	TiO_2	Cr_2O_3	Mn_2O_3	P_2O_5	ZnO	Cl	L.O.I*
1	0.069	0.363	0.005	0.155[1]	0.070	0.010	0.020[2]	0.24
2	0.069[1]	0.365	0.006[1]	0.159	0.090	0.012	0.025[2]	0.25
3	0.070	0.368	0.006	0.160	0.090	0.014	0.030[2]	0.26
4	0.070	0.370	0.007	0.167	0.090	0.015	0.033[2]	0.28
5	0.070	0.370	0.007	0.170	0.090	0.017	0.034[2]	0.29
6	0.070	0.370	0.008	0.170	0.090		0.036[2]	0.32
7	0.070	0.371	0.010	0.170	0.091		0.040[2]	0.33
8	0.070	0.373		0.170	0.094		0.044[3]	0.37
9	0.070	0.375		0.173	0.095		0.049[2]	0.38
10	0.071	0.380		0.173	0.100		0.057[2]	0.42
11	0.074	0.380		0.176	0.110		0.067[2]	0.47
12				0.190				0.48
13								0.49
Mean	**0.070**	**0.371**	**0.007**	**0.169**	**0.092**	**0.014**	**0.040**	**0.35***
Std Dev	0.001	0.005	0.002	0.009	0.010	0.003	0.014	0.09
$C_{(95\%)}$	0.001	0.004	0.002	0.006	0.006	0.003	0.009	0.05

***LOI values have been revised, see below.**

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 test results.

The analytical data listed above are based on original sample material tested in a proficiency test held in 2009. A routinely test beginning 2012 resulted in the fact that the LOI value has changed during the 3 years of stockage. Therefore all certified concentrations from 2009 were recalculated with the original LOI on ignited base to be independent of LOI.

In February 2012 10 sealed bottles were opened, regrinded and tested for LOI (1h at 950°C):

Bottle	LOI
1	0.42
2	0.47
3	0.53
4	0.54
5	0.56
6	0.59
7	0.62
8	0.64
9	0.73
10	0.84
Mean	**0.59**
Std Dev	0.12

Participating Laboratories

Fluxana GmbH & Co.KG	Kleve, Germany
Portlandzementwerk Wittekind	Erwitte, Germany
Heidelberg Cement	Ennigerloh, Germany
Lafarge QDSA	Johannesburg, South Africa
Lafarge Soetenich	Soetenich, Germany
Holcim Deutschland AG	Laegerdorf, Germany
Holcim Schweiz AG	Wuerenlingen, Switzerland
Lafarge CTS North America	Montreal, Canada
Lafarge Ciment (Romania)	TARGU-JIU, Romania
Lafarge TCEA	Lyon, France
Lafarge CTEC	Lyon, France
FH Nürnberg Fachbereich Werkstofftechnik	Nürnberg, Germany
VDZ	Düsseldorf, Germany
LERM	Arles, France
Wilhelm Dyckerhoff Institut	Wiesbaden, Germany
MYKOLAIVCEMENT	MYKOLAIV, Ukraine
Onigbolo plant	COTONOU, Benin

Analytical Methods Used

Most results shown on page 4 and 5 were derived by fused-bead XRF to DIN EN ISO 29581-2: 2007. Results marked with a superscript ([2, 3] etc), were derived by methods involving dissolution, as follows:

1 ICP-AES

2 EN-196-2 methods for S^{2-} (titration with iodine), Cl (titration with NH_4SCN), SO_4^{2-} (gravimetry), Al_2O_3, CaO, Fe_2O_3 and MgO (after chemical separation and volumetric assessment using EDTA), SiO2 (gravimetry and photometry), Na_2O and K_2O (flame photometry).

3 XRF after sample preparation as pressed pellet.

4 SO_3 calculated after combustion with infra-red detection.

Notes

This Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO 17043, ISO Guide 34-2009, ISO Guide 31-2000 and ISO Guide 35-2006.

This certification is applicable to the whole of the sample.

As-supplied, this material will not remain stable indefinitely. The matrix will be affected by contact with the atmosphere, and in particular it will absorb moisture. However, it continues to be fit for use for an indeterminate period, on the understanding that the sample will be ignited prior to weighing, bead preparation and measurement.

All production records will be retained for a period of 10 years from the date of this certificate. This certification will therefore expire in **February 2032**, although we reserve the right to make changes as issue revisions, in the intervening period.

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

The original certification of this product was performed by C Eveleigh, PhD, Technical Director, MBH Analytical Ltd, Barnet, UK in 2009, the recertification in February 2012 was done by R. Schramm, PhD, Director, Fluxana GmbH & Co. KG, Kleve, Germany.