

CERTIFICATE OF ANALYSIS

FLX-CRM 100

Reference Material Information

Type: Cement
Form and Size: Granulate, as-produced, 50g
Manufactured by: Heidelberg Cement Ennigerloh, Germany
Packaged and tested by: Fluxana Dr. Rainer Schramm, Germany
Certified by: MBH Analytical Limited, UK

Assigned Values

Percentage element by weight

Constituent	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	P ₂ O ₅	SO ₃	SiO ₂
Value ¹	5.54	64.51	2.62	0.82	1.47	0.166	2.97	20.89
Uncertainty ²	0.03	0.14	0.01	0.11	0.02	0.006	0.03	0.06

Constituent	SrO	TiO ₂	Cr ₂ O ₃	Mn ₂ O ₃	ZnO	Na ₂ O	Cl	L.O.I
Value ¹	0.286	0.283	0.009	0.066	0.051	0.23	(0.09)	2.37
Uncertainty ²	0.003	0.004	0.002	0.004	0.002	0.02	-	0.14

Notes: all values (except LOI) apply after ignition at 950C for 1 hour.
values given in parentheses are not certified - they are provided for information only.

Definitions

- ¹ The above values are the present 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, detailed on page 3.
- ² The uncertainty values are generated from the 95% confidence interval derived from the analysis results, in combination with a statistical assessment of the homogeneity data.

Certified by:

P/P Fluxana GmbH & Co

on 9th March 2009


C Eveleigh

Tel. +49 2821 973875 / Fax +49 2821 973876 www.fluxana.com

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.

Tel. +49 2821 973875 / Fax +49 2821 973876 www.fluxana.com

Persönlich haftender Gesellschafter Fluxana Verwaltungs-GmbH Sitz in Kleve Geschäftsführer Dr. Rainer Schramm Amtsgericht Kleve HR-A 2935 HR-B 8211 Ust-IdNr.: DE 814692564 Steuer-Nr. 116/5755/0442 Finanzamt Kleve

Analytical Data

Percentage element by weight

Sample	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	P ₂ O ₅	SO ₃	SiO ₂
1	5.45*	64.25	2.57	0.67	1.44	0.159	2.86*	20.74*
2	5.51	64.29	2.61	0.70	1.45	0.160	2.88	20.78
3	5.52	64.36	2.61	0.75	1.46	0.160	2.93	20.82
4	5.54	64.43	2.61	0.75	1.46	0.160	2.95	20.85
5	5.54	64.43	2.61	0.77*	1.46*	0.162	2.97	20.87
6	5.54	64.43	2.61*	0.81	1.47	0.162	2.97	20.89
7	5.54	64.45	2.62	0.84	1.47	0.163	2.98	20.92
8	5.55	64.53	2.62	0.84	1.47	0.170	2.99	20.93
9	5.55	64.57	2.62	0.86	1.48	0.177	2.99	20.93
10	5.55	64.58	2.62	0.87	1.48	0.185*	3.00	20.93
11	5.56	64.60	2.63	0.89	1.49		3.00	20.94
12	5.57	64.60	2.64	0.89	1.49		3.02	20.95
13	5.58	64.66	2.65	0.89	1.50		3.03	20.98
14	5.61	64.90*	2.65	0.90	1.52		3.06	20.98
Mean	5.54	64.51	2.62	0.82	1.47	0.166	2.97	20.89
Std Dev	0.04	0.17	0.02	0.08	0.02	0.009	0.05	0.07
C _(95%)	0.02	0.10	0.01	0.04	0.01	0.006	0.03	0.04

Sample	SrO	TiO ₂	Cr ₂ O ₃	Mn ₂ O ₃	ZnO	Na ₂ O	Cl	L.O.I
1	0.280	0.272	0.005	0.060	0.048	0.20	0.09*	2.21
2	0.282	0.278	0.006	0.063	0.050	0.21		2.24
3	0.282	0.279	0.008	0.064	0.051	0.22*		2.26
4	0.285	0.280	0.009	0.064	0.052	0.23		2.34
5	0.288	0.283	0.010	0.064	0.052	0.23		2.44
6	0.290	0.285	0.010	0.066	0.053	0.23		2.49
7	0.290	0.286	0.010	0.067	0.053	0.23		2.63
8	0.290*	0.290	0.010*	0.070		0.24		
9		0.290		0.080*		0.25		
10		0.290*				0.25		
11						0.27		
Mean	0.286	0.283	0.009	0.066	0.051	0.23	(0.09)	2.37
Std Dev	0.004	0.006	0.002	0.006	0.002	0.02	-	0.15
C _(95%)	0.003	0.004	0.002	0.004	0.002	0.01	-	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 test results.

Participating Laboratories

Fluxana GmbH & Co.KG
Lafarge Ciment (Romia) S.A.
Lafarge Asia
Lafarge Perlmooser GmbH
Lafarge Zement Karsdorf
Lafarge Beocinska Fabrika Cementa
Lafarge TCEA
Lafarge Contes
Holcim Deutschland AG
FH Nürnberg Fachbereich Werkstofftechnik
Nasicecement d.d
Portlandzementwerk Wittekind
VDZ
LERM

Kleve, Germany
Hoghiz, Romania
Chemor, Malaysia
Mannersdorf, Austria
Kall, Germany
Beocin, Serbia
Lyon, France
Contes, France
Sehnde, Germany
Nürnberg, Germany
Nasice, Croatia
Erwitte, Germany
Düsseldorf, Germany
Arles, France

Analytical Methods Used

Most results shown on page 3 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_3) was analysed gravimetrically as BaSO_4 ; chloride (Cl^-) was analysed volumetrically using thiocyanate after Ag precipitation. Other elements were analysed by ICP-AES.

Notes

This Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, taking into account 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.

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 March 2019, 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 certification of this product was performed by C Eveleigh, PhD, Technical Director, MBH Analytical Ltd, Barnet, UK.

Tel. +49 2821 973875 / Fax +49 2821 973876 www.fluxana.com