

Certificate of Analysis

FLX-CRM 111, FLX-CRM 112

Reference Material Information

Type:	2 different refractories
Form and Size:	Granulate, as-produced, ca. 80g each bottle
Manufactured by:	HUK Umweltlabor GmbH, Germany
Packaged and tested by:	FLUXANA GmbH & Co.KG, Germany
Certified by:	FLUXANA GmbH & Co.KG, Germany

Description

Together with HUK Umweltlabor GmbH, Germany 2 new refractories were selected. In May 2011 in total 80kg were supplied.

The samples were taken, grinded and homogenized by HUK to guarantee a fresh homogenized material. The complete batch was sealed into 50ml bottles in May 2011.

In detail the following refractories were taken:

CRM 111	Chrommagnesite
CRM 112	Alumina with Zirconoxide

Certified values and their uncertainties

	FLX-CRM 111		FLX-CRM 112	
	Value ¹	Uncertainty ²	Value ¹	Uncertainty ²
Al2O3	4,66	0,095	79,81	0,360
CaO	2,07	0,035	0,147	0,016
Fe2O3	9,54	0,091	0,326	0,039
HfO2	< 0,01		0,099	0,018
K2O	0,010	0,004	0,090	0,004
MgO	70,20	0,717	0,755	0,042
Na2O	< 0,1		0,267	0,038
NiO	0,031	0,004	< 0,01	
SiO2	1,40	0,065	12,16	0,147
SO3	(0,13)		(0,04)	
TiO2	0,160	0,003	0,273	0,013
ZrO2	0,057	0,006	5,95	0,201
Co3O4	0,012	0,001	< 0,01	
WO3	< 0,02		0,041	0,015
Cr2O3	11,48	0,340	0,017	0,003
Mn3O4	0,370	0,030	0,024	0,006
P2O5	0,088	0,005	0,074	0,008
LOI*	(0,58)		(5,42)	

Notes: all values (except LOI) apply after ignition at 1025°C until constant mass.

*LOI @ 1025°C is informal only and is excluded from certification because it might change over time.

Definitions

- ¹ The above percentages element by weight 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 $C(95\%) = (t \times s) / \sqrt{n}$ where t is the appropriate Student's value, n the number of acceptable mean values and s the standard deviation.

Certified by

Fluxana GmbH & Co.KG



on 16th October 2012

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 50ml bottles with ca. 80g content. These materials are normally used as refractories.

Intended use

Calibration and control sample for x-ray fluorescence (xrf) analysis according EN ISO 12677:2011.

Instructions for the correct use of the CRM

This material has to be ignited at 1025°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 re-ignition might be necessary. The minimum sample quantity for analysis should be 0.5g. The material is moisture sensitive.

For XRF use, ignited samples should be prepared as a fused bead, using e.g. 1 part sample + 8 parts Lithium tetraborate, prepared on an automated fusion machine, and otherwise in accordance with EN ISO 12677:2011.

Hazardous situation

Not classified as dangerous according 67/548/EEC, 1999/45/EC and directive 1272/2008/EC (CLP).

Level of homogeneity

The batch was checked for uniformity using a wavelength-dispersive XRF unit, and an inhouse test method.

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).

For the use as calibration and control sample it has to be noticed that for FLX-CRM 111 the fusion precision determined in the FLUXANA laboratory of Al₂O₃, Fe₂O₃ and ZrO₂ and for FLX-CRM 112 the fusion precision Fe₂O₃ exceeds the allowed tolerance limit f_T of EN ISO 12677:2011.

Calculated tolerances according EN ISO 12677:2011 compared with fusion precision determined by FLUXANA

	FLX-CRM 111		FLX-CRM 112	
	f_T ISO 12677:2011	Fusion precision ¹	f_T ISO 12677:2011	Fusion precision ¹
Al ₂ O ₃	0,036	0,050	0,272	0,141
CaO	0,015	0,011	0,005	0,005
Fe ₂ O ₃	0,049	0,052	0,006	0,012
HfO ₂			0,005	0,002
K ₂ O	0,005	0,005	0,005	0,005
MgO	0,252	0,104	0,015	0,012
Na ₂ O			0,022	0,008
NiO	0,005	0,002		
SiO ₂	0,025	0,011	0,074	0,045
SO ₃				
TiO ₂	0,005	0,003	0,006	0,004
ZrO ₂	0,005	0,008	0,033	0,015
Co ₃ O ₄	0,005	0,001		
WO ₃			0,005	0,004
Cr ₂ O ₃	0,058	0,049	0,005	
Mn ₃ O ₄	0,006	0,005	0,005	
P ₂ O ₅	0,005	0,003	0,005	
LOI				

¹ According EN ISO 12677:2011 fusion precision is the standard deviation of the concentration value of 6 repeated fused bead preparations from the same bottle.

Traceability

The analytical work performed to assess this material has been carried out by competent, laboratories, from institutes, raw material, steel and cement industry. 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.

Values obtained by individual laboratories or methods

Please see the detailed report from the proficiency test for this information.

Methods used

The method most used is x-ray fluorescence analysis with fused bead as sample preparation based on EN ISO 12677:2011.

A few laboratories used:

x-ray fluorescence analysis with fused bead as sample preparation,
x-ray fluorescence analysis with fused bead as sample preparation and reconstitution,
x-ray fluorescence analysis with pressed pellet as sample preparation,
wet chemical methods with digestion and ICP-OES for the determination of oxides,
combustion technique to detect total sulfur.

Loss on ignition (L.O.I) was determined by gravimetry at 1025°C according EN ISO 12677:2011.

Further information

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 and carbon dioxide. 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 15.10.2022, although we reserve the right to make changes as issue revisions, in the intervening period.

The certification, packaging, analysis and storage of this product were supervised by Dr. Rainer Schramm, General Manager, Fluxana GmbH & Co. KG, Bedburg-Hau, Germany.

Participating Laboratories

Bachema AG	Schlieren	Switzerland
BASF Coatings GmbH	Münster	Germany
CRB Analyse Service GmbH	Hardeggen	Germany
Deutsches Institut für Feuerfest und Keramik GmbH	Bonn	Germany
Dorfner Anzaplan	Hirschau	Germany
FLUXANA GmbH & Co.KG	Bedburg-Hau	Germany
Fundacion ITMA	Llanera-Asturias	Spain
Grothe Rohstoffe GmbH & Co. KG	Bückerburg	Germany
Holcim (Deutschland) AG	Sehnde	Germany
Hoppecke Batterien GmbH & Co. KG	Brilon-Hoppecke	Germany
HuK Umweltlabor GmbH	Wenden-Hünsborn	Germany
Imerys Minerals Ltd.	Cornwall	England
Instituto Nacional del Carbón (INCAR-CSIC)	Oviedo (Asturias)	Spain
Rockwool BV	JG Roermond	Netherlands
ThyssenKrupp Steel Europe AG	Duisburg	Germany
Vargön Alloys AB	Vargön	Sweden