

# **CERTIFICATE OF ANALYSIS**

**New certificate issued September 2016** 

# FLX-CRM 128 Feldspar

Mass fraction in %	Certified value <sup>1)</sup>	Uncertainty <sup>2)</sup>	Traceable to
Al <sub>2</sub> O <sub>3</sub>	19.95	0.10	SI unit kg/kg
ВаО	0.007	0.003	Info only
CaO	1.08	0.05	SI unit kg/kg
Fe <sub>2</sub> O <sub>3</sub>	0.021	0.008	SI unit kg/kg
K₂O	0.206	0.009	SI unit kg/kg
Na2O	10.74	0.02	SI unit kg/kg
P2O5	0.008	0.003	Info only
SiO <sub>2</sub>	67.88	0.64	SI unit kg/kg
SrO	0.049	0.010	SI unit kg/kg
TiO <sub>2</sub>	0.017	0.004	Info only
LOI (1h 950°C)	0.171	0.025	Info only

- 1) Certified value traceable to SI unit kg/kg based on dried sample material for 1 hour at 105°C.
- 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 100.12%. This includes LOI.

Bedburg-Hau, 28.09.2016

3. Solatu

**Responsible Reference Materials** 

Dr. Barbara Schäfer

**Statistics and Report** 

Dr. Rainer Schramm

XRF Application Solutions

### **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 40g bottles. This material is normally used as RAW Material, e.g for ceramics.

### 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 has to be dried for minimum 1 hour at 105°C prior use. The drying process must result in a constant weight. 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 or ISO 12677:2011.

The following table contains the certified values *conc<sub>original</sub>* recalculated to ignited sample values *conc<sub>ignited</sub>* as obtained directly from, e.g., a fused bead by the following formula:

$$conc_{ignited} = \frac{conc_{original} * Sum}{Sum - LOI}$$

FLX-CRM 128	Certified values for ignited sample			
mass%	CONCignited	U <sub>CRM</sub>	Traceable to	
Al <sub>2</sub> O <sub>3</sub>	19.98	0.10	SI unit kg/kg	
BaO	0.007	0.003	Info only	
CaO	1.08	0.05	SI unit kg/kg	
Fe <sub>2</sub> O <sub>3</sub>	0.021	0.008	SI unit kg/kg	
K <sub>2</sub> O	0.206	0.009	SI unit kg/kg	
Na₂O	10.75	0.02	SI unit kg/kg	
P <sub>2</sub> O <sub>5</sub>	0.008	0.003	Info only	
SiO <sub>2</sub>	67.99	0.64	SI unit kg/kg	
SrO	0.049	0.010	SI unit kg/kg	
TiO <sub>2</sub>	0.017	0.004	Info only	
LOI				
Sum	100.12			

### **Expiration of Certification**

This certificate is valid, within the uncertainty specified, **until 28.09.2026**. 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.

XRF Application Solutions

#### **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  $u_{hh}^2$ .

$$u^{2}_{bb} = \frac{MS_{among} - MS_{within}}{n}$$

 $MS_{among}$  $MS_{within}$ n quadratic mean of the results of homogeneity between bottle quadratic mean of the results of homogeneity within bottle 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<sup>2</sup>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 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 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 synthetic RMs. The error of the calibration used can be neglected because only the differences in the results between the new reference material and the synthetic RM are part of the evaluation. Synthetic RMs are made from pure chemicals by weighing.

Measurement method used: XRF fusion method for RAW materials

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### **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.

For comparison with the certified values. the results of an independently performed proficiency test are also given. A detailed report is available at <a href="https://www.fluxana.com">www.fluxana.com</a>.

FLX-CRM 128	Traceable to		For Comparison	
	SI unit kg/kg		results of PT	
mass%	X <sub>CRM</sub>	U <sub>CRM</sub>	$X_{PT}$	U <sub>PT</sub>
Al <sub>2</sub> O <sub>3</sub>	19.95	0.10	20.04	0.09
ВаО			0.007	0.003
CaO	1.08	0.05	1.10	0.03
Fe <sub>2</sub> O <sub>3</sub>	0.021	0.008	0.030	0.009
K <sub>2</sub> O	0.206	0.009	0.202	0.003
Na₂O	10.74	0.02	10.75	0.14
P <sub>2</sub> O <sub>5</sub>			0.008	0.003
SiO <sub>2</sub>	67.88	0.64	67.81	0.25
SrO	0.049	0.010	0.048	0.004
TiO <sub>2</sub>			0.017	0.004
LOI			0.171	0.025
Sum			100.19	

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



# **CERTIFICATE OF ANALYSIS**

# FLX-CRM 129 Feldspar

Mass fraction in %	Certified value <sup>1)</sup>	Uncertainty <sup>2)</sup>	Traceable to
Al <sub>2</sub> O <sub>3</sub>	16.44	0.06	SI unit kg/kg
ВаО	0.130	0.007	SI unit kg/kg
CaO	0.374	0.009	SI unit kg/kg
Fe <sub>2</sub> O <sub>3</sub>	0.104	0.007	Info only
K <sub>2</sub> O	10.78	0.02	SI unit kg/kg
Na2O	2.11	0.11	SI unit kg/kg
P2O5	0.068	0.004	SI unit kg/kg
SiO <sub>2</sub>	69.84	0.69	SI unit kg/kg
SrO	0.014	0.002	Info only
TiO <sub>2</sub>	0.036	0.007	Info only
LOI (1h 950°C)	0.428	0.053	Info only

- 1) Certified value traceable to SI unit kg/kg based on dried sample material for 1 hour at 105°C.
- 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 100.33%. This includes LOI.

Bedburg-Hau, 16.06.2016

3. Solati

**Responsible Reference Materials** 

Dr. Barbara Schäfer

Statistics and Report

Dr. Rainer Schramm

Your supplier for XRF Application Solutions

### **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 40g bottles. This material is normally used as RAW Material, e.g for ceramics.

### 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 has to be dried for minimum 1 hour at 105°C prior use. The drying process must result in a constant weight. 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 or ISO 12677:2011.

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$$conc_{ignited} = \frac{conc_{original} * Sum}{Sum - LOI}$$

FLX-CRM 129	Certified values for ignited sample			
mass%	CONCignited	U <sub>CRM</sub>	Traceable to	
Al <sub>2</sub> O <sub>3</sub>	16.52	0.06	SI unit kg/kg	
ВаО	0.131	0.007	SI unit kg/kg	
CaO	0.374	0.009	SI unit kg/kg	
Fe <sub>2</sub> O <sub>3</sub>	0.104	0.007	Info only	
K <sub>2</sub> O	10.83	0.02	SI unit kg/kg	
Na₂O	2.12	0.11	SI unit kg/kg	
P <sub>2</sub> O <sub>5</sub>	0.068	0.004	SI unit kg/kg	
SiO <sub>2</sub>	70.14	0.69	SI unit kg/kg	
SrO	0.014	0.002	Info only	
TiO <sub>2</sub>	0.036	0.007	Info only	
LOI				
Sum	100.33			

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Your supplier for XRF Application Solutions

#### **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  $u_{hh}^2$ .

$$u^{2}_{bb} = \frac{MS_{among} - MS_{within}}{n}$$

 $MS_{among}$   $MS_{within}$  n

quadratic mean of the results of homogeneity between bottle quadratic mean of the results of homogeneity within bottle 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<sup>2</sup>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.

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Measurement method used: XRF fusion method for RAW materials

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### **Further information**

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FLX-CRM 129	Traceable to		For Comparison	
	SI unit kg/kg		results of PT	
mass%	X <sub>CRM</sub>	U <sub>CRM</sub>	$X_{PT}$	U <sub>PT</sub>
Al <sub>2</sub> O <sub>3</sub>	16.44	0.06	16.55	0.11
ВаО	0.130	0.007	0.135	0.008
CaO	0.374	0.009	0.371	0.038
Fe <sub>2</sub> O <sub>3</sub>			0.104	0.007
K <sub>2</sub> O	10.78	0.02	10.66	0.08
Na₂O	2.11	0.11	2.11	0.03
P <sub>2</sub> O <sub>5</sub>	0.068	0.004	0.070	0.003
SiO <sub>2</sub>	69.84	0.69	70.04	0.25
SrO			0.014	0.002
TiO <sub>2</sub>			0.036	0.007
LOI			0.428	0.053
Sum			100.52	

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