

# CERTIFICATE OF ANALYSIS

## ERM<sup>®</sup>-ED105

Yttrium Stabilized Zirconium Oxide		
Parameter	Mass fraction	
	Certified Value <sup>1)</sup> in mg/kg	Uncertainty <sup>2)</sup> in mg/kg
Aluminium	660	15
Calcium	242	9
Iron	95	9
Magnesium	12.9	1.7
Silicon	195	40
Thorium	112	17
Titanium	497	11
Uranium	292	19
	Certified Value <sup>1)</sup> in %	Uncertainty <sup>2)</sup> in %
Hafnium	1.535	0.024
Yttrium	6.11	0.09

1) The certified values are the means of 11-20 series of results (depending on the parameter) obtained by different laboratories. Up to 7 different analytical methods were used for the measurement of each parameter. The methods applied for determination of element mass fractions were calibrated using pure substances of definite stoichiometry or solutions prepared from them, thus achieving traceability to the International System of Units (SI).

2) The uncertainty of the certified value is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) with a coverage factor  $k = 2$ . It includes contributions from sample inhomogeneity.

This certificate is valid until 09/2035; this validity may be extended as further evidence of stability becomes available.

### Sample description

The reference material ERM<sup>®</sup>-ED105 consists of an yttrium stabilized zirconium oxide powder. The material is supplied in glass bottles containing 47 g each.

### Note

European Reference Material ERM<sup>®</sup>-ED105 was produced and certified under the responsibility of Bundesanstalt für Materialforschung und -prüfung (BAM) in cooperation with the Committee of Chemists of the GDMB, Gesellschaft der Metallurgen und Bergleute e. V., according to the principles laid down in the technical guidelines of the European Reference Materials<sup>®</sup> co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (<http://www.erm-crm.org>).

Accepted as an ERM<sup>®</sup>, 23.07.2015

BAM Department 1  
Analytical Chemistry;  
Reference Materials

BAM Division 1.6  
Inorganic Reference Materials

Prof. Dr. U. Panne  
(Head of Department)

Dr. S. Recknagel  
(Head of Division)

Additional Material Information		
Parameter		Mass fraction <sup>1)</sup> in mg/kg
Phosphorus		< 75
1) The value stated is based on 5 series of results obtained by different laboratories. Two different analytical methods were used for the measurement of the parameter.		
Parameter		Mass fraction in %
Phase <sup>2)</sup>	monoclinic	1.94
Particle size distribution <sup>3)</sup>		Particle size in $\mu\text{m}$
	d <sub>10</sub>	18.9
	d <sub>50</sub>	33.3
	d <sub>90</sub>	55.4
2) The measurements were carried out by X-ray powder diffraction using Rietveld method for evaluation.		
3) The particle size distribution was determined by laser light diffraction method.		

### Recommendations for Correct Sampling and Sample Preparation

To ensure representative sub-sampling for the analysis the CRM bottle should be shaken in different directions for about two minutes before taking the sub-sample. According to the sub-sample mass used for the homogeneity testing the minimum sample intake for analysis is 200 mg. It is not required to dehydrate the sample before starting measurements.

### Participating laboratories

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany  
 Bruker AXS GmbH, Germany  
 BCRC, Mons, Belgium  
 ESK Ceramics GmbH & Co. KG, Kempten, Germany  
 Forschungsinstitut für anorganische Werkstoffe - Glas/Keramik GmbH, Höhr-Grenzhausen, Germany  
 Forschungszentrum Jülich, Jülich, Germany  
 GfE Fremat GmbH, Freiberg, Germany  
 H. C. Starck GmbH & Co. KG, Werk Goslar, Goslar, Germany  
 H. C. Starck GmbH & Co. KG, Werk Laufenburg, Laufenburg, Germany  
 Institute Jozef Stefan, Ljubljana, Slovenia  
 Karlsruher Institut für Technologie, Karlsruhe, Germany  
 Leibniz Institut für Kristallzüchtung, Berlin, Germany  
 Max-Planck-Institut für intelligente Systeme, Stuttgart, Germany  
 Osram AG, Augsburg, Germany  
 Plansee SE, Reutte, Austria  
 Revierlabor GmbH, Essen, Germany  
 Treibacher Industrie AG, Treibach-Althofen, Austria  
 Umicore AG & Co. KG, Hanau, Germany  
 Wolfram Bergbau und Hütten AG, St. Martin i.S., Austria

## Laboratory means

line no.	Al [mg/kg]	Ca [mg/kg]	Fe [mg/kg]	Mg [mg/kg]	Si [mg/kg]	Th [mg/kg]	Ti [mg/kg]	U [mg/kg]	Hf [%]	Y [%]	P [mg/kg]	ZrO <sub>2</sub> (monoclinic) [%]
1	614.3	---	71.8	9.5	80.8	60.3	---	235.7	1.469	5.780	< 4	1.8
2	627.5	219.3	73.3	9.5	97.8	90.5	454.0	251.8	1.474	5.984	< 10	2.0
3	635.0	223.8	79.5	9.7	132.3	91.3	463.0	280.5	1.481	5.992	< 20	2.01
4	638.3	230.3	80.9	10.0	150.3	95.3	473.7	283.6	1.482	5.998	48.3	
5	641.7	231.7	82.4	10.2	151.7	112.6	474.0	295.3	1.485	6.020	71.9	
6	648.3	235.0	82.7	10.9	152.2	114.7	485.0	297.2	1.497	6.024		
7	649.0	236.7	83.3	11.3	186.8	117.8	494.7	298.2	1.518	6.092		
8	653.5	238.3	83.3	11.3	192.0	121.5	497.9	300.8	1.537	6.093		
9	655.5	239.8	83.6	11.4	202.8	130.0	498.3	301.7	1.539	6.098		
10	656.7	240.7	85.8	12.0	204.1	131.0	499.8	333.3	1.540	6.118		
11	660.2	241.2	89.4	12.5	206.2	166.7	500.2	335.8	1.543	6.125		
12	665.0	243.7	89.7	12.7	206.3		501.7		1.554	6.127		
13	671.2	244.7	95.5	13.2	254.3		506.7		1.558	6.148		
14	676.6	246.7	103.3	13.3	272.5		508.3		1.560	6.155		
15	695.0	271.6	107.3	16.0	299.0		516.0		1.564	6.207		
16	704.3	280.0	111.7	18.3	326.7		525.0		1.567	6.237		
17	726.3	---	117.3	20.0	---		526.7		1.625	6.360		
18	---		119.8	20.2			530.0		1.630	6.368		
19			131.7						---	---		
20			137.5									
M:	659.9	241.6	95.5	12.9	194.8	112.0	497.3	292.2	1.535	6.107	< 75	1.94
s <sub>M</sub> :	28.5	15.9	19.3	3.5	68.5	27.7	21.9	29.8	0.048	0.139		

Note: The „ --- “ indicates that an outlying value has been detected by a statistical test and was withdrawn or removed  
Values given in *italic type* are for information only.

M: Arithmetic mean of the laboratory means

s<sub>M</sub>: Standard deviation of the laboratory means

## Analytical methods used for determination

Element	Line No.	Analytical method used
Al	1, 2, 4, 6, 7, 8, 9, 10, 12, 13, 14, 15, 16, 17	ICP-OES
	3	XRF
	5	F AAS
	11	DC Arc OES
Ca	2, 3, 5, 6, 7, 8, 9, 10, 11, 13, 14, 15, 16	ICP-OES
	4	F AAS
	12	XRF
Fe	1, 2, 5, 7, 8, 10, 11, 12, 13, 14, 15, 16, 19, 20	ICP-OES
	3	XRF
	4	ETV-ICP-OES
	6	ICP-MS
	9, 18	F AAS
	17	DC Arc OES
Mg	1, 2, 3, 6, 7, 8, 9, 11, 12, 13, 14, 16, 17	ICP-OES
	4	ETV-ICP-OES
	5	ICP-MS
	10, 18	F AAS
	15	DC Arc OES

Element	Line No.	Analytical method used
Si	1, 2, 3, 4, 7, 9, 10, 11, 12, 14	ICP-OES
	5	Photometry
	6	ETV-ICP-OES
	8	ET AAS
	13	SS ET AAS
	15	XRF
	16	DC Arc OES
Th	1, 3, 9	ICP-OES
	2, 4, 5, 7, 10	ICP-MS
	6	k <sub>0</sub> -INAA
	8	ETV-ICP-OES
	11	XRF
Ti	2, 3, 5, 6, 7, 8, 9, 10, 11, 12, 14, 16, 17, 18	ICP-OES
	4	ETV-ICP-OES
	13, 15	XRF
U	1, 2, 4, 5, 11	ICP-MS
	3, 6, 9	ICP-OES
	7	ETV-ICP-OES
	8	k <sub>0</sub> -INAA
	10	XRF
Hf	1, 2, 4, 6, 7, 8, 9, 10, 11, 14, 15, 17, 18	ICP-OES
	3, 12, 13, 16	XRF
	5	k <sub>0</sub> -INAA
Y	1, 2, 3, 4, 6, 7, 8, 9, 10, 12, 14, 15, 17, 18	ICP-OES
	5, 11, 13, 16	XRF

The measurements of the monoclinic phase of yttrium stabilized zirconium oxide were carried out by X-ray powder diffraction using Rietveld method for evaluation. The tetragonal and cubic phases could not be determined due to signal overlapping.

#### List of abbreviations

DC Arc OES	Direct current arc optical emission spectrometry
ET AAS	Electrothermal atomic absorption spectrometry
ETV-ICP-OES	Inductively coupled plasma optical emission spectrometry with electrothermal vaporisation
F AAS	Flame atomic absorption spectrometry
k <sub>0</sub> -INAA	k <sub>0</sub> -Instrumental neutron activation analysis
ICP-MS	Inductively coupled plasma mass spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
SS ET AAS	Solid sampling electrothermal atomic absorption spectrometry
XRF	X-ray fluorescence spectrometry

#### Intended Use and Recommendations for Correct Storage

This reference material is intended for the calibration of analytical instruments and to validate or verify analytical methods suitable for the analysis of zirconium oxide materials. The sample should be stored in a dust-free and dry environment at room temperature (20 °C).

This Reference Material is offered by:

Bundesanstalt für Materialforschung und -prüfung (BAM)  
Richard-Willstätter-Str. 11, D-12489 Berlin

Phone: +49 30 8104 2061  
Fax: +49 30 8104 1117

Email: [sales.crm@bam.de](mailto:sales.crm@bam.de)  
Internet: [www.webshop.bam.de](http://www.webshop.bam.de)