in cooperation with the WG 'Special Materials'
of the Committee of Chemists of GDMB

European Reference Materials

Certified Reference Material

ERM®-ED102
Boron Carbide Powder

Certified Values			
	Certified value [1]		**Uncertainty** [2]
Parameter	**Mass fraction in mg/kg**		
Aluminium	157	±	5
Calcium	97	±	8
Cobalt	0.39	±	0.09
Chromium	5.6	±	1.2
Copper	2.2	±	0.4
Iron	686	±	22
Manganese	10.4	±	0.5
Sodium	6.3	±	0.9
Nickel	8.0	±	1.6
Silicon	268	±	22
Titanium	96	±	5
Zirconium	48.9	±	2.3
	Mass fraction in %		
Total Carbon	21.01	±	0.28
Oxygen	0.10	±	0.04
Nitrogen	0.209	±	0.026
Total Boron [3]	78.47	±	0.31
Soluble Boron [4]	0.116	±	0.013
Boron Oxide [5]	0.075	±	0.023
	Isotopic abundance in %		
^{10}Boron [6]	19.907	±	0.014

1) The certified values are the means calculated from the laboratory means of 7- 24 sets of single values (depending on the parameter) which were reported by the participating laboratories. Between 2 and 8 different analytical methods were used for the measurement of each parameter. The calibration of the methods applied for determination of element mass fractions was carried out by using pure substances of known stoichiometry or by solutions prepared from them, thus achieving traceability to the SI unit.
2) The uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurements (GUM) with a coverage factor k = 2. It includes contributions from sample inhomogeneity and from potential deterioration of the sample until the expiration of the validity of the certificate.
3) The recommended "Method M1" described in attachment can be used for the determination of total mass fraction of boron.
4) The recommended "Method M2" described in attachment can be used for the determination of mass fraction of in HNO_3 soluble boron.
5) The recommended "Method M3" described in attachment can be used for the determination of mass fraction of boron oxide.
6) Isotopic abundance (amount fraction) of ^{10}Boron related to total amount of Boron.

Sample description and intended use

The certified reference material ERM®-ED102 consists of a boron carbide powder (type 305F422). The material is supplied in glass bottles containing 100 g each. The reference material is intended for use in the calibration of analytical instruments or to validate or verify analytical methods to be used for the determination of the certified parameters in boron carbide. The material can also be used to calibrate analytical instruments or to validate or verify analytical methods used for the determination of the total carbon mass fraction in other materials having similar carbon contents.

Indicative values

Non certified, indicative values are given for additional analytes determined in the interlaboratory comparison by participating laboratories. They are given as indicative values, because the spread of values obtained was considerably larger than can be accepted for certified values.

	Indicative value [1)]		Uncertainty [2)]
Parameter	**Mass fraction in mg/kg**		
Magnesium	3.2	±	1.0
Tungsten	3.6	±	2.1
	Mass fraction in %		
Free Carbon [3)]	0.51	±	0.12

1) Indicative values are the means of 5-18 series of results (depending on the parameter) obtained by different laboratories. Between 1 and 4 different analytical methods have been used for the measurement of each parameter. The methods applied for the determination of mass fraction were not calibrated in all cases by pure substances of known stoichiometry or by solutions prepared from them.
2) The uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurements (GUM) with a coverage factor k = 2. The values are quoted for information purposes.
3) The prescribed “Method M4” described in attachment shall be used for the determination of mass fraction of free carbon.

Additional Material Information

Additional material properties were determined by using one method, and can be used as informative values only.

		Particle size in µm
Particle size [1)]	D_{10}	21.5
	D_{50}	33.6
	D_{90}	51.4
	D_{97}	60.4

1) The particle size distribution (volume) was determined by laser light diffraction method. Terms D_{xy} according to ISO 9276-1.

European Reference Material ERM®-ED102 was certified under the responsibility of Bundesanstalt für Materialforschung und -prüfung (BAM) in cooperation with the Committee of Chemists of the GDMB, Gesellschaft für Bergbau, Metallurgie, Rohstoff- uns Umwelttechnik according to the principles laid down in the technical guidelines of the European Reference Material ERM® cooperation agreement between BAM-LGC-IRMM. Information on these guidelines is available in the Internet (http://www.erm-crm.org)

Accepted as an ERM®, Berlin, November 18 2008. Validity of the Certificate: Until June 30 2015.

BAM Berlin

Department I
Analytical Chemistry; Reference Materials

Division I.1
Inorganic Chemical Analysis; Reference Materials

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

Dr. D. Lück
(p. p. Head of Division)

Laboratory means *)

Mass fractions – arranged in increasing value

Result no.	Al [mg/kg]	Ca [mg/kg]	Co [mg/kg]	Cr [mg/kg]	Cu [mg/kg]	Fe [mg/kg]	Mn [mg/kg]	Na [mg/kg]	Ni [mg/kg]	Si [mg/kg]	Ti [mg/kg]
1	-	61	0.28	4.0	1.4	-	8.1	5.3	6.2	-	-
2	141	62	0.30	4.5	1.5	599	9.4	5.4	6.2	205	90
3	143	83	0.31	4.7	1.6	630	9.6	5.6	6.4	216	90
4	145	87	0.39	4.7	1.7	646	9.7	5.7	6.6	227	90
5	152	89	0.41	4.7	1.7	646	9.7	5.9	7.1	238	91
6	152	90	0.42	5.2	2.0	650	9.9	6.4	7.2	264	92
7	153	91	0.45	5.2	2.3	665	10.0	6.8	7.4	265	92
8	153	91	0.45	5.4	2.3	666	10.1	7.0	7.5	275	93
9	154	92	0.53	5.4	2.7	669	10.2	7.1	7.8	281	93
10	155	93	-	5.4	2.8	669	10.3	7.6	7.8	292	94
11	155	93	-	5.5	2.8	673	10.4	-	8.3	295	95
12	156	94		5.7	3.0	679	10.6		10.0	295	95
13	158	96		5.7	3.2	687	10.8		10.1	304	96
14	158	96		5.7	-	689	10.8		10.6	323	96
15	159	97		6.9		689	10.9		11.1	-	97
16	159	99		7.8		692	11.0			-	97
17	160	103		9.3		692	11.0				97
18	163	105		-		695	-				98
19	163	107		-		696	11.4				101
20	164	110				709	11.7				102
21	168	115				720	11.9				104
22	173	135				763	12.7				104
23	177	135				771					105
24						792					
M:	157	97	0.39	5.6	2.2	686	10.4	6.3	8.0	268	96
s_M:	9	18	0.09	1.3	0.7	45	1.0	0.8	1.7	37	5

*) Some laboratories delivered more than one set of results coming from different methods applied

The ' - ' indicates that an outlying value has been detected by a statistical test which was withdrawn or omitted after discussion in GDMB meetings.

Note: The result number does not relate to the laboratory code number

M: Arithmetic mean of the laboratory means

s_M: Standard deviation of the laboratory means (rounded up)

Laboratory means *)

Part 2

Mass fractions and isotopic abundance(for $^{10}B/(^{10}B+^{11}B)$) *arranged in increasing value*

Result no.	Zr [mg/kg]	C_{total} [%]	O [%]	N [%]	B_{total} [%]	$B_{soluble}$ [%]	B_2O_3 [%]	$^{10}B/(^{10}B+^{11}B)$ **) [%]	*Mg* [mg/kg]	*W* [mg/kg]	C_{free} [%]
1	37.3	20.5	0.067	0.172	-	0.098	0.056	19.880	*1.3*	*1.1*	*0.39*
2	44.7	20.6	0.080	0.186	78.09	0.112	0.057	19.897	*1.4*	*1.2*	*0.44*
3	44.8	20.8	0.081	0.187	78.11	0.113	0.066	19.901	*1.4*	*5.0*	*0.45*
4	45.5	20.8	0.083	0.200	78.16	0.117	0.067	19.905	*1.7*	*5.1*	*0.60*
5	47.2	20.9	0.089	0.204	78.17	0.118	0.073	19.908	*1.7*	*5.6*	*0.66*
6	47.5	20.9	0.091	0.206	78.23	0.121	0.078	19.922	*1.7*		
7	47.7	20.9	0.100	0.219	78.25	0.137	0.082	19.938	*1.7*		
8	48.0	20.9	0.106	0.221	78.25	-	0.084	-	*2.2*		
9	48.7	20.9	0.109	0.224	78.25	-	0.108		*2.3*		
10	49.2	20.9	0.114	0.226	78.38				*2.6*		
11	49.5	21.0	0.118	0.230	78.46				*2.8*		
12	50.0	21.0	0.122	0.233	78.68				*2.9*		
13	50.3	21.0			78.76				*3.5*		
14	50.4	21.0			78.80				*4.7*		
15	50.7	21.0			78.81				*5.6*		
16	51.3	21.0			78.99				*6.3*		
17	51.4	21.1			79.06				*6.7*		
18	54.1	21.2							*7.3*		
19	54.5	21.2									
20	55.4	21.3									
21	-	21.5									
22		21.6									
23											
24											
25											
M:	48.9	21.0	0.097	0.209	78.47	0.116	0.075	19.907	*3.2*	*3.6*	*0.51*
s_M:	4.0	0.3	0.018	0.020	0.33	0.012	0.017	0.019	*2.0*	*2.3*	*0.12*

*) Some laboratories delivered more than one set of results coming from different methods applied

The ' - ' indicates that an outlying value has been detected by a statistical test which was withdrawn or omitted after discussion in GDMB meetings.

Values given in *italic type* are indicative values only.

Note: The line number does not relate to the laboratory code number

**): Isotopic abundance (amount fraction) of ^{10}Boron related to total amount of Boron

M: Arithmetic mean of the laboratory means

s_M: Standard deviation of the laboratory means (rounded up)

Analytical methods used for final determination

List of abbreviations

CGHE-Coul.	Carrier gas hot extraction method by coulometric determination
CGHE-IR	Carrier gas hot extraction method with infrared detection
CGHE-TC	Carrier gas hot extraction method with thermal conductivity detection
Comb.-Coul.	Combustion of total carbon followed by coulometric determination
Comb.-Grav.	Combustion of total carbon followed by gravimetric determination
Comb.-IR	Combustion method with infrared detection
Comb.-Vol.	Combustion of total carbon followed by volumetric determination
Coul.	Coulometric determination
DC-ARC-OES	Direct current arc optical emission spectrometry
ET AAS	Atomic absorption spectrometry with electrothermal atomization
ETV-ICP OES	Inductively coupled plasma optical emission spectrometry with electrothermal vaporisation
F AAS	Flame atomic absorption spectrometry
ICP OES	Inductively coupled plasma optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ID-ICP-MS	Isotope dilution inductively coupled plasma mass spectrometry
IPAA	Instrumental photon activation analysis
MAS	Molecular absorption spectrometry
Method M1 *)	Recommended Method: Determination of Total Boron in Boron Carbide by Titrimetric Method (potentiometric method)
Method M2 *)	Recommended Method: Determination of HNO_3-soluble Boron in Boron Carbide by Titrimetric Method
Method M3 *)	Recommended Method: Determination of Adherent Boron Oxide in Boron Carbide by Titrimetric Method
Method M4 *)	Prescribed Method: Determination of Free Carbon in Boron Carbide by wet Chemical Oxidation
SS ET AAS	Solid sampling electrothermal atomic absorption spectrometry
TIMS	Thermal ionization mass spectrometry
TITR	Titrimetry

*) The Methods M1 –M4 are described in the Appendix of the Certification Report which can be found at BAM-website

Element	**Result No.** in the Table above (Laboratory Means)	**Analytical method used**
Al	(1), 9	DC-ARC-OES
	7	ET AAS
	3	ETV-ICP OES
	4, 18	ICP-MS
	2, 5, 6, 8, 10, 11, 12, 13, 14, 15, 16, 17, 19, 20, 21, 22, 23	ICP OES
Ca	2, 15	DC-ARC-OES
	17	ETV-ICP OES
	10, 11	F AAS
	13, 22	ICP-MS
	1, 3, 4, 5, 6, 8, 9, 12, 14, 16, 18, 19, 20, 21, 23	ICP OES
	7	IPAA
Co	4	ET AAS
	2	ETV-ICP OES
	1, 5, 8	ICP-MS
	6, 7, 9, (10)	ICP OES
	3	IPAA

Element	**Result No.** in the Table above (Laboratory Means)	**Analytical method used**
Cr	(18)	DC-ARC-OES
	2	ET AAS
	4	ETV-ICP OES
	1, 5, 10	ICP-MS
	3, 6, 7, 8, 9, 11, 12, 13, 14,15, 16, 17, (19)	ICP OES
Cu	3	DC-ARC-OES
	5	ET AAS
	2	ETV-ICP OES
	1, 4, 9	ICP-MS
	6, 7, 8, 10, 11, 12, 13, (14)	ICP OES
Fe	(1), 19	DC-ARC-OES
	5	ETV-ICP OES
	14, 20	F AAS
	10, 11	ICP-MS
	2, 3, 4, 7, 8, 9, 12, 13, 16, 17, 18, 21, 22, 23, 24	ICP OES
	15	IPAA
	6	MAS
Mg	17	DC-ARC-OES
	2	ET AAS
	5	ETV-ICP OES
	6, 11, 15	ICP-MS
	1, 3, 4, 7, 8, 9, 10, 12, 13, 14, 16, 18	ICP OES
Mn	1	DC-ARC-OES
	6	ET AAS
	2	ETV-ICP OES
	5, 8	ICP-MS
	3, 4, 7, 9, 10, 11, 12, 13, 14, 16, 17, (18), 19, 20, 21, 22	ICP OES
	15	IPAA
Na	2	ET AAS
	5	ETV-ICP OES
	1, 3, 6, 7, 10	F AAS
	4	ICP-MS
	8, 9, (11)	ICP OES
Ni	15	DC-ARC-OES
	8	ET AAS
	2	ETV-ICP OES
	1, 3	ICP-MS
	4, 5, 6, 7, 9, 10, 11, 12, 13, 14	ICP OES
Si	(1), 13	DC-ARC-OES
	3	ET AAS
	14	ETV-ICP OES
	4, (16)	ICP-MS
	2, 5, 6, 9, 10, 11	ICP OES
	7, 8, 12, (15)	MAS
Ti	21	DC-ARC-OES
	18	ET AAS
	(1)	ETV-ICP OES
	9, 20	ICP-MS
	2, 3, 4, 5, 6, 7, 8, 10, 11, 13, 14, 15, 16, 17, 19, 22, 23	ICP OES
	12	IPAA
W	1, 2, 5	ICP-MS
	3, 4	ICP OES

Element	**Result No.** in the Table above (Laboratory Means)	**Analytical method used**
Zr	19	DC-ARC-OES
	1	ETV-ICP OES
	5, 7	ICP-MS
	2, 3, 4, 6, 8, 9, 10, 11, 12, 13, 15, 16, 17, 18, 20, (21)	ICP OES
	14	IPAA
C_{total}	3, 6	Comb.-Coul.
	14	Comb.-Grav.
	2, 4, 5, 7, 8, 9, 10, 11, 12, 13, 15, 16, 17, 18, 19, 20, 21, 22	Comb.-IR
	1	Comb.-Vol.
C_{free}	4	Coul.
	1, 2, 3, 5	Wet Chem. Oxidation-Coul. (Method M4)
O	5	CGHE-Coul.
	1, 2, 3, 4, 6, 7, 8, 9, 10, 11, 12	CGHE-IR
N	1, 2, 3, 4, 5, 6, 7, 8, 9, 11, 12	CGHE-TC
	10	IPAA
B_{total}	12, 16	ICP OES
	(1)	ID-ICP-MS
	2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 13, 14, 15, 17	TITR (Method M1)
$B_{soluble}$	2, 4, 7	ICP OES
	1, 3, 5, 6, (8), (9)	TITR (Method M2)
B_2O_3	6, 7, 8	ICP OES
	1, 2, 3, 4, 5, 9	TITR (Method M3)
$B^{10}/(B^{10}+B^{11})$	2, 3, 4, 7, (8)	ICP-MS
	1, 5, 6	TIMS

Line numbers in parenthesis refer to values not used in the final calculation of the certified value

Participating laboratories (arranged alphabetically)

Asahi Glass Ceramics Co., LTD, Development Center, Japan
Bundesanstalt für Materialforschung und -prüfung, Germany
- Division I.1
- Division I.4

Ceram Testing & Environmental Services, U.K.
Dunhua Zhengxing Abrasive Co., Ltd., P.R. China
Eagle Picher Technologies Boron Dept., USA
ESK Ceramics GmbH Co. KG, Germany
ESK-SiC GmbH, Abteilung MQ, Germany
EUROPÄISCHE KOMMISSION Gemeinsame Forschungsstelle, Institut für Transurane, Germany
Ferro GmbH, Germany
Forschungszentrum Jülich GmbH, Zentralabt. für chemische Analysen, Germany
Framatome ANP GmbH, Abt. NGTR, Germany
H. C. Starck GmbH & Co. KG; Germany
- Werk Goslar
- Werk Laufenburg

HORIBA, Ltd., Application Center, Japan
Japan Analyst Corporation, Japan
JFE Refractories R & D Laboratory, Japan
Johannes Gutenberg Universität Mainz; Institut für Kernchemie, Germany
Krosaki Harima Co., LTD., Technical Examination Center, Japan

Leibnitz-Institut für Festkörper- und Werkstoffforschung, Germany
Max-Planck-Institut für Metallforschung, Germany
NSL Analytical Services, Inc., USA
Osram GmbH, Germany
Plansee AG , Werkanalytik, Austria
PTB, Physikalisch Technische Bundesanstalt, Germany
Revierlabor Chemische Laboratorien für Industrie und Umwelt GmbH, Germany
Rigaku Industrial Corp., Japan
SGL Carbon GmbH , Laboratory Services, Germany
Shanghai Institute of Ceramics, Chinese Academy of Sciences, P.R. China
Shinagawa Refractories Co., LTD., Testing & Evaluation Center, Japan
Taiko Refractories Co., LTD, Research & Development Laboratory Japan
Treibacher Industrie AG, Austria
TYK Corporation, Research & Development Center, Japan
Verein für Kernverfahrenstechnik und Analytik Rossendorf e.V., Germany
Zhuzhou Cemented Carbide Group Corp., LTD., P.R. China

Recommendations for Correct Sampling and Sample Preparation

To ensure a representative sub-sampling for the analysis the bottle containing the CRM should be shaken in different directions for about two minutes before taking the sub-sample. Each sub-sample has to be taken separately. According to the different sub-sample masses for the homogeneity testing different minimum sub-sample masses are specified for different analytes (in parenthesis /mg): Al, Ca, Co, Cr, Cu, Fe, Mg, Mn, Ni, Ti, W, Zr(250); Na, Si(10), C_{total}(25); O, N(50), C_{free}, B_{total}(100); $B_{soluble}$, B_2O_3(4000). The opening duration of the bottle should be as short as possible. The lid of the bottle containing a special sealing gasket should be locked tightly immediately after usage. For subsequent elemental analysis the sample has to be treated thermally at (135 ± 5) °C for 12 hours to achieve defined starting conditions. The pressure digestion procedure used before the determination of metallic analytes has to be checked to ensure that no analyte losses occur during the procedure.

Recommendations for Correct Storage

The sample should be stored in a dust-free and dry environment at room temperature (about 15 °C – 25 °C) avoiding contamination and moisture. No special cooling of the sample is necessary.

Expiration of Certification

The date of expiry of certification is ten years after the date of interlaboratory comparison for certification. Before this date (June 30, 2015) a new certificate will be prepared with a new date of expiry, if necessary.

Safety Guidelines

1. First aid measures
   In the event of contact with the skin, rinse off with water and soap. After contamination of the eyes, they must be rinsed immediately with plenty of water. Seek medical advice in case of continuous irritation.
   If product is swallowed and in case of sickness seek medical advice. The product is not known to be toxic.
2. Accidental release measures
   Precautionary measures regarding persons: Avoid formation and deposition of dust. Ensure effective ventilation.
   Methods for cleaning up / taking up: Take up mechanically; avoid dust formation. Fill into labelled, sealable containers.
3. Handling
   Avoid formation and deposition of dust. Ensure adequate ventilation and if necessary, exhaust ventilation when handling or transferring the product. Keep away from sources of ignition and do not smoke. Fine dust may form explosive mixture with air. Powder with particle size < 10 µm: Substance is rated to dust explosion class ST 1 according to German VDI 2263.
4. Exposure restriction and personal protection
   Do not smoke when handling. Do not breathe dust.
   Respiratory protection:
   Respirator fine mask with filter typ P1 according to DIN EN 143. Hand protection: not required. Eye protection: protective goggles
5. Limit values of dust concentration in air to be monitored
   Regulatory instructions concerning limit values of concentration of different particle size are to be maintained.
6. Disposal considerations
   May be disposed of in approved special landfills provided local regulations are observed.

Regulatory and Material Information

This information is part of the certification report which is uploaded at the BAM website.