BAM Bundesanstalt für Materialforschung und -prüfung

in co-operation with the Committee of Chemists of the GDMB
Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik

Certified Reference Material

BAM-S008

Silicon Carbide Powder (transparent 200/F)

Certified Values

Parameter	Mass fraction [1] in mg/kg	Uncertainty [2] in mg/kg
Aluminium	47	7
Boron	3.0	1.2
Calcium	0.25	0.06
Chromium	0.16	0.05
Copper	0.10	0.05
Iron	4.8	0.8
Magnesium	0.07	0.07
Manganese	0.05	0.02
Sodium	0.17	0.09
Nickel	0.9	0.5
Titanium	67	6
Vanadium	275	18
Zirconium	4.4	1.2
Nitrogen	18	4
Oxygen	146	36
	Mass fraction [1] in %	**Uncertainty [2] in %**
$Carbon_{total}$	29.9	0.1
$Carbon_{free}$	0.045	0.010

1) The certified values are the means of 6-17 series of results (depending on the parameter) obtained by different laboratories. 2 up to 8 different analytical methods were used for the measurement of one 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 SI unit.
2) The certified 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.

Sample Description

The certified reference material BAM-S008 consists of silicon carbide powder (type transparent 200/F). The material is supplied in glass bottles containing 50 g each.

Indicative Values

Parameter	Mass fraction [1] in mg/kg
Silicon dioxide	< 0.01
$Silicon_{free}$	< 0.03

1) The indicative values are estimated from 3 series of results each obtained by different laboratories.

Informative Values

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

Parameter		Mass fraction in %	Uncertainty in mass %
Phases:	SiC-6H	99.7 [1]	0.2 [2]
	SiC-15R	0.23 [1]	0.2 [2]
	SiC-4H	0.06 [1]	0.2 [2]
Particle size distribution		**Size distribution in µm**	
	d_{10}	24.3 [3]	
	d_{50}	65.4 [3]	
	d_{90}	125.3 [3]	

1) The analyses were carried out by high resolution powder diffraction using synchrotron radiation (λ = 0.08 nm) and the Rietveld method for the evaluation of the diffraction data.
2) The calculation of the standard uncertainty is based on the evaluation of the diffraction data of ten specimens by the Rietveld method.
3) The particle size distribution was determined by laser light diffraction method.

Recommended Use

The main area of application is checking the trueness of results when one or more of the certified parameters in silicon carbide material are determined by a laboratory. Based on own results and on certified values the uncertainty of own measurements can be calculated. The material can also be used for checking the trueness of the determination of the total carbon content in other refractory materials having similar carbon mass fractions. The material may be used for phase identification and quantification of coarsely grained silicon carbide and other ceramic ‘powders‘ as well.

Means of Accepted Data Sets

Line-No.	Al [mg/kg]	B [mg/kg]	Ca [mg/kg]	Cr [mg/kg]	Cu [mg/kg]	Fe [mg/kg]	Mg [mg/kg]	Mn [mg/kg]	Na [mg/kg]	Ni [mg/kg]	Ti [mg/kg]	V [mg/kg]	Zr [mg/kg]	N [mg/kg]	O [mg/kg]	C_{total} [%]	C_{free} [%]
1	40.2	1.43	0.126	0.088	0.065	3.89	0.028	0.033	0.102	0.495	-	210	2.58	12.7	78.2	29.57	0.035
2	41.3	2.40	0.143	0.121	0.078	4.35	0.029	0.045	0.107	0.495	46.3	232	2.65	15.7	92.3	29.65	0.036
3	42.6	2.95	0.237	0.125	0.091	4.63	0.039	0.047	0.127	0.532	53.3	249	3.00	18.0	100.0	29.72	0.036
4	44.3	3.33	0.265	0.127	0.095	4.72	0.055	0.047	0.163	0.577	54.5	265	3.83	18.5	104.0	29.73	0.040
5	44.6	3.96	0.289	0.149	0.097	4.72	0.056	0.050	0.167	0.863	61.2	269	4.18	19.8	121.7	29.82	0.044
6	45.7	4.18	0.290	0.152	0.103	4.77	0.110	0.052	0.213	0.908	63.7	271	4.20	22.5	124.4	29.86	0.061
7	50.2		0.317	0.155	0.150	4.78	0.167	0.055	0.229	0.940	70.0	273	4.40		163.2	29.91	0.062
8	50.4		0.332	0.233		4.94	-	0.064	0.262	1.044	70.8	286	4.71		168.2	29.94	
9	50.5			0.282		5.05		0.064		1.200	71.4	288	5.24		194.3	29.94	
10	52.2			-		5.13		-		1.667	72.7	291	5.37		219.0	29.95	
11	52.7					5.17		-			73.8	295	5.42		245.5	29.98	
12	55.3					5.33					73.8	298	5.44			30.00	
13						-					74.4	308	5.64			30.00	
14						-					76.0	317				30.01	
15						-					76.5	-				30.03	
16											-					30.04	
17																30.15	
18																-	
M:	47.5	3.04	0.250	0.159	0.097	4.79	0.069	0.051	0.171	0.872	67.0	275	4.36	17.9	146.4	29.90	0.045
s_M:	5.0	1.02	0.077	0.061	0.027	0.39	0.051	0.010	0.059	0.374	9.6	29	1.08	3.4	55.5	0.16	0.012

The „ - “ indicates that an outlying value has been detected by a statistical test which was withdrawn or omitted
Note: The line number should not be mistaken for the laboratory code number.
M: Arithmetic mean of the laboratory means
s_M: Standard deviation of the laboratory means

Analytical Methods

Element	Line No.	
Al	1, 4, 7, 8	ICP-OES
	2, 3, 10, 12	DCarc-OES
	5	ICP-SF-MS
	6	ET AAS
	9	GD-MS
	11	XRF
B	1, 4	ETV-ICP-OES
	2, 6	GD-MS
	3	DCarc-OES
	5	ICP-SF-MS
Ca	1, 2, 4	ETV-ICP-OES
	3	ICP-MS
	5	ET AAS
	6	ICP-SF-MS
	7	ICP-OES
	8	GD-MS
Cr	1	INAA
	2	ICP-SF-MS
	3	ET AAS
	4, 5, 6, 7	ETV-ICP-OES
	8	ICP-OES
	9	GD-MS
Cu	1, 5	ETV-ICP-OES
	2, 6	GD-MS
	3	ET AAS
	4	ICP-SF-MS
	7	ICP-OES
Fe	1, 2, 6, 12	ETV-ICP-OES
	3, 4	GD-MS
	5	DCarc-OES
	7	INAA
	8	ICP-SF-MS
	9	ICP-OES
	10	ET AAS
	11	ICP-MS
Mg	1, 3, 4, 5	ETV-ICP-OES
	2	ET AAS
	6	GD-MS
	7	ICP-OES
Mn	1, 4	ETV-ICP-OES
	2, 7	GD-MS
	3, 8	INAA
	5	ET AAS
	6	ICP-SF-MS
	9	ICP-MS

Element	Line No.	
Na	1	ICP-SF-MS
	2, 5	ET AAS
	3	SS ET AAS
	4	INAA
	6	GD-MS
	7, 8	ETV-ICP-OES
Ni	1, 2, 3, 10	ETV-ICP-OES
	4, 6	GD-MS
	5	ICP-SF-MS
	7	ET AAS
	8	ICP-MS
	9	ICP-OES
Ti	2, 14	GD-MS
	3	ETV-ICP-OES
	4, 9, 10, 13, 15	ICP-OES
	5, 6, 7, 12	DCarc-OES
	8	ET AAS
	11	ICP-SF-MS
V	1, 13	GD-MS
	2, 3	ETV-ICP-OES
	4	INAA
	5, 6, 7, 9	DCarc-OES
	8, 11, 12, 14	ICP-OES
	10	ET AAS
Zr	1, 2, 3, 5	ETV-ICP-OES
	4, 8, 12, 13	ICP-OES
	6, 11	GD-MS
	7, 9	DCarc-OES
	10	ICP-SF-MS
C_{total}	1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 15, 17	Comb.-IR
	7, 16	Comb./coul.
C_{free}	1, 4, 5, 7	Comb.-IR
	2, 3	wet chem. oxidation/coul. (Method M1)
	6	Comb./coul.
O	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	CGHE
N	1, 2, 3, 4, 5, 6,	CGHE

List of Abbreviations

CGHE	Carrier gas hot extraction
Comb./coul.	Combustion method followed by coulometric determination
Comb.-IR	Combustion method with infrared detection
DCarc-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

F AES	Flame atomic emission spectrometry
GRAV	Gravimetry
GD-MS	Glow discharge mass spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ICP-SF-MS	Inductively coupled plasma sector field mass spectrometry
INAA	Instrumental neutron activation analysis
Method M1	Coulometric determination after wet chemical oxidation with hot chromic sulphuric acid (described in report)
SS ET AAS	Solid sampling electrothermal atomic absorption spectrometry
TITR	Titrimetry
Vol.	Volumetric determination
XRF	X-ray fluorescence spectrometry

Participating Laboratories

BAM Bundesanstalt für Materialforschung und -prüfung Berlin (Germany)
- Laboratory: Activation Analysis; Gas Analysis
- Laboratory: Inorganic Chemical Analysis; Reference Materials

Chinese Academy of Sciences, Shanghai Institute of Ceramics, Shanghai (PR China)
ESK Ceramics GmbH Co. KG, Kempten (Germany)
ESK-SiC GmbH, Frechen (Germany)
Forschungszentrum Jülich GmbH, Jülich (Germany)
H.C. Starck GmbH & Co. KG, Goslar (Germany)
H.C. Starck GmbH & Co. KG, Laufenburg (Germany)
Horiba Ltd., Tokyo (Japan)
Japan Fine Ceramics Center, Atsuta-ku Nagoya (Japan)
JEF Refractories, Ako-City, Hyougo (Japan)
Johannes Gutenberg Universität, Institut für Kernchemie, Mainz (Germany)
Krosaki Harima Corp. Ltd., Kitakyusyu (Japan)
Leibniz-Institut für Festkörper- und Werkstoffforschung, Dresden (Germany)
Leibniz-Institut für Kristallzüchtung, Berlin (Germany)
Max-Plank-Institut für Metallforschung, Stuttgart (Germany)
NGK Insulators, LTD., Chemical analysis materials research lab., Nagayo (Japan)
OSRAM GmbH, München (Germany)
Rigaku Industrial Corp., Takasuki-shi, Osaka (Japan)
Shiva Technologies Inc., Evans Analytical Group, Tournefeuille (France)
SGL Carbon GmbH, Bonn (Germany)
SGL Carbon GmbH, Meitlingen (Germany)
TYK Corp., Research and Development Center, Tajimi-City (Japan)
Umicore Precious Metals Refining, Hoboken (Belgium)

Handling

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: Al, Ca, Cr, Cu, Mg, Na, Ni, Ti, V, Zr (10 mg); B, Fe, Mn (15 mg); C_{total} (18 mg); C_{free} (200 mg); O, N (100 mg); Si_{free}, SiO_{2free} (500 mg). 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 the determination of metallic analytes, the required pressure digestion has to be tested concerning the loss of analytes and completeness of the decomposition.

Transport and Storage

The sample should be stored in a dust-free and dry environment.

Safety Guidelines

For detailed information to safety guidelines and handling of the material, please see the Material Safety Data sheet distributed by the producer of the material (available on request).

Literature

A. Dette, S. Richter, B. Peplinski, S. Recknagel, Certification Report BAM-S008

ISO Guide 31, Contents of certificates of reference materials, 1981

ISO Guide 34, General requirements for the competence of reference material producers, 2009

ISO Guide 35, Reference materials - General and statistical principles for certification. Third edition, 2006

Guidelines for the production of BAM Reference Materials, 2006

Bonas G, Zervou M, Papaeoannou T, Lees M: Accred Qual Assur (2003) 8:101-107

Guide to the Expression of Uncertainty in measurement (1995), International Organization for Standardization, ISBN 92-67-10188-9

Peplinski B, Fitch AN, Evans A, Ibberson RM, Többens DM, Crabswick LMD, Dörfel I, Emmerling F, Matschat R: Z. Kristallogr. Suppl. 30 (2009) 61-66

Regulatory Information

- ANSI American National Standards Institute, Methods of chemical analysis of silicon carbide abrasive grain and abrasive crude, ANSI B74.15-1992, ANSI American National Standards Institute, 1992, pp. 20
- DIN, ISO 51079-1, Prüfung keramischer Roh- und Werkstoffe, Chemische Analyse von Siliciumcarbid als Rohstoff und als Bestandteil von Werkstoffen, Teil 1 Soda-Borsäure-Aufschluss
- DIN, ISO 51079-2, Prüfung keramischer Roh- und Werkstoffe, Chemische Analyse von Siliciumcarbid als Rohstoff und als Bestandteil von Werkstoffen, Teil 2 Säure-Druck-Aufschluss
- DIN, ISO 51079-3, Prüfung keramischer Roh- und Werkstoffe, Chemische Analyse von Siliciumcarbid als Rohstoff und als Bestandteil von Werkstoffen, Teil 3 Aufschluß des freien Kohlenstoffs durch naßchemische Oxidation
- DIN, ISO, 51075 - 1-5, Prüfung keramischer Roh- und Werkstoffe, Chemische Analyse von Siliciumcarbid, Teil 1-5

- FEPA (Féderation Européenne des Fabricants de Produits Abrasifs), Chemische Analyse von Siliciumcarbid, FEPA-Standard 45-D-1986, Fachverband Elektrokorund- und Siliziumkarbid-Hersteller e.V., Verein Deutscher Schleifmittelwerke e.V., 1986, 1-25

Accepted as BAM-CRM on 01-10-2013

BAM Bundesanstalt für Materialforschung und -prüfung

Prof. Dr. U. Panne
Head of Department 1
Analytical Chemistry;
Reference Materials

Dr. S. Recknagel
Head of Division 1.6
Inorganic Reference Materials

This Reference Material is offered by:

BAM Bundesanstalt für Materialforschung und -prüfung
Richard-Willstätter-Straße 11, 12489 Berlin

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

Email: sales.crm@bam.de
Internet: www.webshop.bam.de