Bundesanstalt für Materialforschung und -prüfung (BAM)

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

Certified Reference Material

BAM-S003a

Silicon Carbide Powder (green micro F 800)

Please notice the safety guidelines (see Page 7)

Element/ Constituent	Certified Values		
	Symbols	Mass fraction [1] in mg/kg	Uncertainty [2] in mg/kg
Aluminium	Al	372	20
Boron	B	63	7
Calcium	Ca	29.4	2.7
Chromium	Cr	3.5	0.4
Copper	Cu	1.5	0.4
Iron	Fe	149	15
Magnesium	Mg	6.3	0.9
Manganese	Mn	1.44	0.25
Sodium	Na	17.7	0.8
Nickel	Ni	32.9	2.7
Titanium	Ti	79	4
Vanadium	V	41	5
Zirconium	Zr	25.2	2.0
Free Carbon [3]	C_{free}	493	79
		Mass fraction [1] in %	Uncertainty [2] in %
Total Carbon [4]	C_{total}	29.89	0.07

1) The certified values are the means of 4-22 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 calibration of the methods applied for determination of element mass fractions were carried out by using pure substances of definite stoichiometry or by using 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 (Ca, Fe, Mg, Mn: k = 3). It includes contributions from sample inhomogeneity.
3) The mass fraction of carbon free is a method-depending value. It was determined by two different methods and is only related to the application of these methods, which are described as "Method M1" and "Method M2" respectively, attached to this certificate.
4) The recommended "Method M3" described in attachment can be used for the determination of mass fraction of carbon total.
5) The recommended "Method M4" described in attachment can be used for the determination of mass fraction of oxygen.

Date of certification of BAM-S003: 2004

Sample description

The certified reference material BAM-S003a is a silicon carbide powder (type green micro F800). The material is supplied in glass bottles containing 50 g each. It is based on the same batch of candidate material as BAM-S003. The mass fraction of the element O differs slightly from the original material. Therefore this element is given only for information with a higher uncertainty. All mass fractions are obtained from the certification inter-laboratory comparison of BAM-S003 from 2004.

Indicative values

Not certified indicative values were determined in the interlaboratory comparison by participating laboratories.

Parameter	Indicative Values	
	Mass fraction [1] in mg/kg	Uncertainty [2] in mg/kg
Nitrogen	93	22
Oxygen	910	53
Silicon dioxide free	600	148
Silicon free	481	223

1) The indicative values are the means of 6-11 series of results (depending on the parameter) obtained by different laboratories. 1 up to 4 different analytical methods were used for the measurement of one parameter. The methods applied for determination of mass fractions were not always calibrated by using pure substances of definite stoichiometry or by using solutions prepared from them, thus was not achieved traceability to SI units.
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 $k = 2$ (Oxygen: $k = 3$).

Additional Material Data

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

Parameter		Additional Material Data	
		Mass fraction in %	Uncertainty in mass %
Phases:	SiC-6H	89.2 [1]	0.2 [2]
	SiC-15R	6.1 [1]	0.2 [2]
	SiC-4H	4.7 [1]	0.2 [2]
Parameters of particle size		Particle size in µm	
	D_{10}	5.55 [3]	
	D_{50}	10.18 [3]	
	D_{90}	16.69 [3]	

1) The measurements were carried out by X-ray powder diffraction using Rietveld method for evaluation.
2) The calculation of the standard uncertainty is based on a raw estimation from the evaluation of the Rietveld method.
3) The particle size distribution was determined by laser light diffraction method.

Means of the series of measurements for the analytical procedure of one laboratory (Laboratory means)

mass fractions in mg/kg (C_{total} in mass%)

Serial Nº	Al	B	Ca	Cr	Cu	Fe	Mg	Mn	Na	Ni	Ti	V	Zr	C_{total} (%)	C_{free}	O	*N*	*$SiO_{2\,free}$*	*Si_{free}*
1	-	55	21.2	2.5	1.0	-	3.8	1.00	-	24.1	-	-	19.1	-	415	825	*47*	*488*	*117*
2	305	55	25.4	2.6	1.1	-	5.5	1.10	15.0	24.2	-	28.83	21.2	-	500	845	*59*	*552*	*398*
3	327	61	-	2.6	1.2	131	5.8	1.30	15.3	26.2	68	30.83	22.0	29.57	515	862	*64*	*583*	*400*
4	334	62	27.4	3.2	1.2	135	5.9	1.32	15.7	28.7	70	36.29	22.4	29.75	540	865	*80*	*608*	*468*
5	345	63	29.1	3.3	1.3	135	5.9	1.33	16.7	29.8	72	37.00	22.8	29.82		873	*89*	*632*	*550*
6	354	63	29.1	3.4	1.3	137	6.0	1.36	17.0	30.5	73	38.00	23.2	29.85		902	*89*	*733*	*950*
7	367	63	29.1	3.4	1.4	137	6.0	1.37	17.3	30.9	74	39.27	23.7	29.86		915	*94*		
8	371	65	29.5	3.5	1.4	140	6.1	1.38	17.7	31.0	77	39.67	23.7	29.87		948	*99*		
9	371	66	29.5	3.5	1.4	142	6.3	1.41	17.8	31.1	77	41.25	24.3	29.89		951	*115*		
10	373	66	29.8	3.6	1.8	143	6.3	1.42	18.0	31.3	77	41.35	25.4	29.90		988	*128*		
11	377	67	29.8	3.6	2.3	143	6.4	1.47	18.7	31.7	77	42.38	25.6	29.91		1032	*151*		
12	378	-	30.5	3.9	2.5	146	6.5	1.48	18.8	32.2	79	42.53	25.8	29.91		-			
13	381	-	32.0	4.0	-	149	6.5	1.49	19.1	32.6	79	44.38	26.0	29.92					
14	385		32.7	4.1		149	6.8	1.53	19.3	33.1	81	45.48	26.6	29.94					
15	392		35.7	4.2		149	6.9	1.72	19.4	35.2	83	45.87	27.0	29.94					
16	399		-	4.4		152	8.2	1.79	19.5	35.5	83	46.07	28.7	29.96					
17	402		-	-		154	9.0	2.03	-	36.5	84	47.49	29.1	29.96					
18	403					155	-	-	-	39.2	84	48.27	30.4	30.03					
19	415					158	-			39.4	85	49.92	31.0	30.13					
20						162				39.4	85		-						
21						164				40.5	87								
22						164				41.1									
23						173													
24						-													
M:	372	63	29.4	3.5	1.5	149	6.3	1.44	17.7	32.9	79	41.38	25.2	29.89	493	910	*93*	*600*	*481*
s_M:	30	5	3.4	0.6	0.5	12	1.1	0.24	1.5	5.0	6	5.74	3.2	0.12	55	65	*31*	*83*	*273*

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 serial 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 used for final determination

List of abbreviations

CGHE/comb.-IR	Carrier gas hot extraction/combustion method with infrared detection
Comb./coul.	Combustion of free carbon 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
ICP OES	Inductively coupled plasma optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
K_0-INAA	K_0- Instrumental neutron activation analysis
MAS	Molecular absorption spectrometry
Method M1	Coulometric determination after wet chemical oxidation with hot chromic sulfuric acid, (described in appendix)
Method M2	Combustion of free carbon followed by coulometric determination comprising weighing back the sample boat, (described in appendix)
SS ET AAS	Solid sampling electrothermal atomic absorption spectrometry
TITR	Titrimetry
Vol.	Gas volumetric determination

Element	Line No.	Analytical method used
Al	4, 12	DCarc-OES
	8	ET AAS
	2	ETV-ICP OES
	(1)	F AAS
	16	ICP-MS
	3, 5, 6, 7, 9, 10, 11, 13, 14, 15, 17, 18, 19	ICP OES
B	3	DCarc-OES
	1, 2, 4, 5, 7, 8, 9, 10, 11, (12), (13)	ICP OES
	6	MAS
Ca	1	ETV-ICP OES
	5, (17)	F AAS
	13	ICP-MS
	2, 3, 4, 6, 7, 8, 9, 10, 11, 12, 14, 15,(16)	ICP OES
Cr	2	DCarc-OES
	9, 16	ET AAS
	3	ETV-ICP OES
	8	ICP-MS
	1, 4, 6, 11, 12, 13, 14, 15, (17)	ICP OES
	7, 10	INAA
	5	K_0-INAA
Cu	11	DCarc-OES
	4	ET AAS
	3	ETV-ICP OES
	9	ICP-MS
	1, 2, 5, 6, 7, 8, 10, 12, (13)	ICP OES

Element	Line No.	Analytical method used
Fe	(1), 5	DCarc-OES
	22	ETV-ICP OES
	9	F AAS
	12	ICP-MS
	(2), 3, 4, 7, 8, 10, 11, 13, 14, 15, 16, 17, 18, 19, 20, 21	ICP OES
	23	INAA
	6	K_0-INAA
	(24)	Titrimetry
Mg	5	DCarc-OES
	9	ET AAS
	12	ETV-ICP OES
	17	F AAS
	15	ICP-MS
	1, 2, 3, 4, 6, 7, 8, 10, 11, 13, 14, 16, (18), (19)	ICP OES
Mn	15	DCarc-OES
	6, 11	ET AAS
	17	ETV-ICP OES
	9	ICP-MS
	1, 2, 4, 8, 10, 12, 13, 14, 16, (18)	ICP OES
	3, 7	INAA
	5	K_0-INAA
Na	(1)	ETV-ICP OES
	5, 7, 11, 16	F AAS
	15	F AES
	8	ICP-MS
	2, 3, 6, 10, 14, (17), (18)	ICP OES
	4, 9	INAA
	12	K_0-INAA
	13	SS ET AAS
Ni	2, 5	DCarc-OES
	7	ET AAS
	1	ETV-ICP OES
	18	F AAS
	10	ICP-MS
	3, 4, 6, 8, 9, 11, 12, 13, 14, 16, 17, 19, 20, 21, 22	ICP OES
	15	K_0-INAA
Ti	(1), 21	DCarc-OES
	4	ETV-ICP OES
	13	ICP-MS
	(2), 3, 5, 6, 7, 8, 9, 10, 11, 12, 14, 15, 16, 17, 18, 20	ICP OES
	19	K_0-INAA
V	2, 16	DCarc-OES
	(1)	ETV-ICP OES
	11	ICP-MS
	3, 4, 5, 6, 7, 8, 9, 10, 12, 13, 14, 15, 18, 19	ICP OES
	17	K_0-INAA
Zr	15	DCarc-OES
	17	ETV-ICP OES
	10, 16	ICP-MS
	1, 2, 3, 4, 5, 6, 7, 8, 9, 12, 13, 14, 19, (20)	ICP OES
	18	INAA
	11	K_0-INAA
C_{total}	(1), 3, 4, 5, 7, 8, 9, 10, 11, 13, 14, 15, 16, 17, 18	CGHE/comb.-IR
	(2)	CGHE/titr.
	6	CGHE/grav.
	12, 19	Coul.

Element	Line No.	Analytical method used
C_{free}	(1), (2), (3), (4), (13), (14)	CGHE/comb.-IR
	(9), (10), (11), (12)	Coul.
	6, 7	Comb./coul. (Method M1)
	5, 8	wet chem. oxidation/coul. (Method M2)
O	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, (12)	CGHE
	11	CGHE/coul.
N	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	CGHE
SiO_{2free}	1	Vol.
	2, 3	MAS
	4, 5	ICP OES
	6	Grav.
Si_{free}	1	Coul.
	2, 4, 5, 6	Vol.
	3	Comb.

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

Participating laboratories (arranged alphabetically)

BAM, Bundesanstalt für Materialforschung und -prüfung Berlin (Germany)
- Laboratory: Activation Analysis; Gas Analysis
- Project group: Quality Assurance and Metrological Aspects in Production of High Tech Reference materials

Chinese Academy of Sciences, Shanghai Institute of Ceramics, Analysis and Testing Center for Inorganic Materials, Shanghai (PR China)
CRB GmbH, Hardegsen (Germany)
DIFK Deutsches Institut für Feuerfest und Keramik GmbH, Bonn (Germany)
Elektroschmelzwerk Delfzijl B.V., TE Farmsum (The Netherlands)
ESK-SiC GmbH, Frechen (Germany)
Forschungszentrum Jülich GmbH, Jülich (Germany)
H.C. Starck Ceramics GmbH & Co. KG; Selb (Germany)
H.C. Starck GmbH & Co. KG; Goslar (Germany)
H.C. Starck GmbH & Co. KG; Laufenburg (Germany)
Institut für Festkörper und Werkstoffforschung, Dresden (Germany)
Japan Fine Ceramics Center, Atsuta-ku Nagoya (Japan)
Johannes-Gutenberg-Universität, Institut für Kernchemie, Mainz (Germany)
Jožef Stefan Institute, Ljubljana (Slovenia)
Max-Plank-Institut für Metallforschung, Stuttgart (Germany)
Molab AS, Mo I Rana (Norway)
NGK Insulators, LTD., Chemical analysis materials research lab., Nagayo (Japan)
OSRAM GmbH, München (Germany)
Plansee AG, Reutte (Austria)
Saint Gobain Ceramic Materials AS, Lillesand (Norway)
Saint Gobain Industrial Ceramics and Plastics, Northboro (USA)
Saint Gobain Industriekeramik, Geschäftsbereich Feuerfesttechnik, Rödental (Germany)
Schunk Kohlenstofftechnik GmbH, Heuchelheim (Germany)
SGL Carbon GmbH, Bonn (Germany)
SGL Carbon GmbH, Meitlingen (Germany)
W.C. Heraeus GmbH, Hanau (Germany)
Wacker Ceramics, Kempten (Germany)
Zhuzhou Cemented Carbide Group Corp., LTD., Zhuzhou, Hunan (PR 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 paranthesis /mg): Al, Ca, Fe, Mg, Ni, Ti, Zr(8); Cr, Cu, V(14); B(500); C_{total}(25); C_{free}(500); O(80). 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. Sample preparation for the determination of the analyte boron has to be carried out by using fusion technique with sodium peroxide followed by an extraction to avoid losses. For subsequent elemental analysis the sample has to be treated thermally at (135 ± 5) °C for 12 hours to achieve defined starting conditions. For the determination of metallic analytes the required pressure digestion has to be verified concerning absence of analyte losses.

Recommendations for Correct Storage

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

Expiration of Certification

The date of expiry of certification is 31st December 2030. Before this date a new certificate will be prepared with a new date of expiry.

Safety Guidelines

1. First aid measures

   In the event of contact with the skin, rinse off with water and soap. Contamination of the eyes must be treated by thorough irrigation with water, with the eyelids held open.
   If product is swallowed, induce vomiting and consult a physician. 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.

4. Exposure restriction and personal protection

   Respiratory protection: If necessary use a respirator mask with filter type P according to DIN EN 143.
   Hand protection: protective gloves
   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

   Unused material: reuse if possible. Address manufacturer of the starting material.
   Or: May be disposed of in approved special landfills provided local regulations are observed.

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

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Bundesanstalt für Materialforschung und -prüfung (BAM)

Dr. S. Richter
Committee for Certification

Dr. S. Recknagel
Project Coordinator

This Reference Material is offered by:

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

Phone: +49 30 8104 2061
Fax: +49 30 8104 72061

E-mail: sales.crm@bam.de
Internet: www.bam.de