

Bundesanstalt für Materialforschung und -prüfung (BAM)

in co-operation with the WG 'Special Materials'
of the Committee of Chemists of GDMB
Gesellschaft der Metallurgen und Bergleute e.V.

Certified Reference Material

BAM-S009

Medium Purity Graphite Powder

Certified Values

Parameter	Mass fraction ¹⁾ in mg/kg	Uncertainty ²⁾ in mg/kg
Al	0.27	0.08
B	0.83	0.18
Ba	0.80	0.09
Be	0.00050	0.00027
Ca	5.1	1.1
Co	0.143	0.017
Cr	1.39	0.20
Cu	0.067	0.012
Fe	28	4
K	1.04	0.20
Li	0.022	0.007
Mg	0.135	0.030
Mn	0.094	0.015
Mo	0.20	0.04
Na	0.32	0.08
Ni	5.6	0.6
P	0.26	0.06
Pb	0.052	0.028
S	10.7	1.8
Si	41	6
Sr	0.32	0.05
Ti	8.6	1.6
V	1.30	0.17
W	3.0	0.6
Y	0.049	0.011
Zn	0.070	0.018
Zr	0.81	0.19

¹⁾ Unweighted mean value of the means of accepted sets of data, each set being obtained by a different laboratory and/or a different method of measurement.

²⁾ Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement, (GUM, ISO/IEC Guide 98-3:2008).

This certificate is valid until 05/2032.

Values for information

Parameter	Mass fraction ¹⁾ in mg/kg	Uncertainty ²⁾ in mg/kg
Ag	0.0018	0.0010
As	0.016	0.007
Bi	0.016	0.012
Cd	0.0022	0.0019
Eu	0.0021	0.0010
Sb	0.022	0.015
Sc	0.012	0.010
Sn	0.16	0.05
Ta	0.018	0.014

¹⁾ Unweighted mean value of the means of accepted sets of data, each set being obtained by a different laboratory and/or a different method of measurement. Values were not certified, but given for information, when the number of accepted data sets were considered to be too low (< 5) or when the uncertainty from the inter-laboratory certification was considerably larger than the expected range or homogeneity data are lacking.

²⁾ Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement, (GUM, ISO/IEC Guide 98-3:2008).

B_{ETV}	0.22 ³⁾	0.09
Si_{ETV}	12.8 ³⁾	2.4

³⁾ Method specific value obtained using ETV-ICP OES with an evaporation temperature of 2300 °C, according to DIN 51457: 2017-05, Testing of ceramic raw and basic materials - Direct determination of mass fractions of trace impurities in powders, granules and lumps of graphite by optical emission spectroscopy by inductively coupled plasma (ICP OES) and by electrothermal vaporization (ETV) under the action of a halogenated reaction gas (modifiers). For further details, see Technical Report.

Further informative values (laboratory mean values without statistical evaluation)										
	Mass fraction in mg/kg									
Line no.	Au	Cs	Dy	Ga	Gd	Hg	In	La	Nb	Rb
1	< 0.00005	0.0038	0.010	< 0.02	0.021	0.0089	< 0.026	0.103	0.11	< 0.02
2	< 0.0032	0.0038	0.012		0.025	0.0089	< 0.05			
3	< 0.07		< 0.07		< 0.1	< 0.031				
4						< 0.06				

(continued)

Line no.	Re	Rh	Se	Sm	Tb	Te	Th	U		Ash
1	< 0.0006	< 0.0055	0.011	0.012	0.0034	0.015	0.011	0.022		202
2	< 0.0086	< 0.4		0.014	0.0037	0.026	0.030	0.037		
3				< 0.3				< 0.1		

Sample Description

The certified reference material BAM-S009 consists of a medium purity graphite powder. The material is supplied in polypropylene bottles containing 40 g each.

Recommended Use

This reference material is intended to be used for the calibration of analytical instruments and for the validation or verification of analytical methods suitable for the analysis of graphite materials. This CRM can be used as multi-element standard for the calibration of solid sampling methods (e.g. ETV-ICP OES, ETV-ICP-MS). Special care must be taken for the calibration of ETV techniques for B and Si.

Means of Accepted Data Sets

Certified values (Mass fraction in mg/kg)

Line no.	Al	B	Ba	Be	Ca	Co	Cr	Cu	Fe	K	Li	Mg	Mn
1	0.116	0.650	---	0.000121	2.80	< 0.07	0.97	0.0417	20.3	0.66	0.0152	0.100	0.0632
2	0.151	0.690	0.557	0.000146	3.09	0.098	1.09	0.0536	21.5	0.75	0.0173	< 0.1	0.0650
3	0.190	0.740	0.605	0.000369	3.48	0.112	1.11	0.0561	21.9	0.79	0.0175	0.100	0.0659
4	0.203	0.753	0.695	0.000647	3.61	0.117	1.11	0.0599	23.9	0.87	0.0195	0.100	0.0767
5	0.206	0.763	0.700	0.000717	3.79	0.117	1.12	0.0607	24.0	0.87	0.0196	0.113	0.0804
6	0.235	0.807	0.733	0.000721	3.88	0.130	1.13	0.0622	24.7	0.93	0.0217	0.118	0.0834
7	0.251	1.098	0.742	0.000792	4.03	0.134	1.16	0.0623	25.2	0.94	0.0222	0.128	0.0845
8	0.254	1.100	0.754		4.35	0.139	1.18	0.0630	25.3	0.95	0.0231	0.130	0.0854
9	0.259		0.774		4.81	0.139	1.26	0.0646	26.2	1.00	0.0274	0.134	0.0924
10	0.260		0.781		4.95	0.140	1.27	0.0666	26.3	1.00	0.0359	0.135	0.0955
11	0.275		0.786		5.11	0.143	1.27	0.0684	27.2	1.04	---	0.135	0.0957
12	0.278		0.786		5.25	0.148	1.29	0.0712	27.2	1.09	---	0.136	0.0970
13	0.285		0.791		5.33	0.150	1.30	0.0746	28.0	1.12		0.136	< 0.1
14	0.286		0.805		5.43	0.150	1.33	0.0760	28.3	1.12		0.136	0.1000
15	0.292		0.834		5.73	0.151	1.35	0.0815	28.4	1.17		0.140	0.1016
16	0.311		0.835		6.52	0.151	1.36	0.1047	28.8	1.24		0.144	0.1026
17	0.314		0.863		6.73	0.152	1.46		29.2	1.41		0.149	0.1045
18	0.350		0.867		6.75	0.155	1.50		29.4	1.44		0.156	0.1074
19	0.362		0.868		6.78	0.155	1.56		30.4	1.44		0.166	0.1114
20	0.462		0.990		7.15	0.158	1.60		31.0			0.171	0.1222
21			0.995		7.78	0.159	1.64		33.3			0.176	0.1533
22			1.126			0.159	1.67		33.4			---	
23			---			0.168	1.74		36.3			---	
24						0.172	1.74		36.7				
25							1.88						
26							1.95						
27							---						
<i>M :</i>	0.267	0.825	0.804	0.000502	5.11	0.143	1.39	0.0667	28.1	1.04	0.0219	0.135	0.0944
<i>s_M :</i>	0.077	0.175	0.127	0.000285	1.44	0.018	0.26	0.0139	4.6	0.22	0.0060	0.022	0.0211

(continued)

Line no.	Mo	Na	Ni	P	Pb	S	Si	Sr	Ti	V	W	Y	Zn	Zr
1	0.075	0.164	4.38	0.132	0.0045	8.0	---	0.265	3.58	1.04	1.93	0.0303	0.0406	0.413
2	0.142	0.178	4.50	0.187	0.0150	8.4	32.8	0.279	4.17	1.10	1.97	0.0403	0.0448	0.537
3	0.147	0.266	4.53	0.222	0.0170	9.4	35.6	0.286	6.51	1.17	2.06	0.0408	0.0467	0.567
4	0.179	0.276	4.55	0.224	0.0181	10.4	37.5	0.292	6.94	1.17	2.12	0.0430	0.0475	0.571
5	0.188	0.279	4.82	0.237	< 0.02	10.5	37.6	0.298	7.10	1.19	2.40	0.0437	0.0581	0.590
6	0.197	0.288	4.83	0.282	0.0358	11.1	40.3	0.306	7.20	1.23	2.47	0.0463	0.0595	0.619
7	0.197	0.288	4.91	0.296	< 0.046	11.4	42.5	0.310	7.43	1.24	2.50	0.0513	0.0603	0.637
8	0.200	0.307	5.10	0.330	0.0555	11.7	42.8	0.313	7.64	1.27	2.50	0.0518	0.0606	0.662
9	0.200	0.318	5.37	0.348	0.0651	12.9	43.6	0.320	7.90	1.30	2.65	0.0590	0.0610	0.689
10	0.202	0.321	5.45	0.376	0.0673	13.1	45.0	0.333	8.43	1.32	2.81	0.0618	0.0637	0.690
11	0.203	0.322	5.50	---	0.0759		45.7	0.336	8.51	1.32	3.08	0.0721	0.0680	0.705
12	0.208	0.324	5.54	---	< 0.1		46.9	0.347	8.85	1.34	3.12		0.0698	0.722
13	0.209	0.338	5.57		0.1059			0.356	8.97	1.35	3.17		0.0717	0.741
14	0.217	0.351	5.59		0.1155			0.363	9.04	1.36	3.34		0.0806	0.792
15	0.230	0.362	5.64		< 0.2			0.375	9.20	1.37	3.43		0.0873	0.826
16	0.263	0.502	5.73		< 0.5			0.401	9.22	1.39	3.86		0.1091	0.845
17	0.337	0.539	5.74						10.13	1.39	4.77		0.1096	0.899
18	---	5.87							10.90	1.49	4.96		0.1275	0.956
19	---	6.07							11.07	1.50				1.013
20		6.25							12.12	1.54				1.090
21		6.29							12.18	---				1.105
22		6.87							12.25	---				1.298
23		7.24												1.572
24		7.30												
<i>M :</i>	0.200	0.319	5.57	0.263	0.0523	10.7	41.0	0.324	8.61	1.30	2.95	0.0491	0.0704	0.806
<i>s_M :</i>	0.054	0.093	0.82	0.077	0.0376	1.7	4.5	0.038	2.31	0.13	0.88	0.0117	0.0242	0.269

Informative values (Mass fraction in mg/kg)

Line no.	<i>Ag</i>	<i>As</i>	<i>Bi</i>	<i>Cd</i>	<i>Eu</i>	<i>Sb</i>	<i>Sc</i>	<i>Sn</i>	<i>Ta</i>	<i>B_{ETV}</i>	<i>Si_{ETV}</i>
1	0.00021	0.0126	0.0018	0.00013	0.0012	0.0037	0.0040	0.099	0.0051	0.101	7.9
2	0.00139	0.0130	0.0119	0.00024	0.0024	0.0038	0.0053	< 0.1	0.0233	0.164	11.0
3	0.00165	0.0150	0.0190	0.00027	0.0027	< 0.004	0.0107	0.139	0.0262	0.176	11.1
4	0.00174	0.0184	0.0300	0.00075	< 0.05	0.0120	0.0116	0.144	< 0.1	0.209	11.7
5	0.00265	< 0.02	< 0.1	0.00147		0.0157	0.0305	0.161		0.209	12.4
6	< 0.003	0.0230		< 0.0015		0.0273		0.186		0.259	12.7
7	< 0.003	---		0.00258		0.0273		0.227		0.290	12.8
8	0.00304			0.00458		< 0.03				0.383	13.3
9	< 0.017			< 0.005		0.0308					13.3
10	< 0.1			0.00719		0.0588		< 0.3			15.0
11				< 0.008				< 0.5			20.1
12				---							
13				< 0.03							
14				< 0.1							
15											
16											
<i>M</i> :	0.00178	0.0164	0.0157	0.00215	0.00208	0.0224	0.0124	0.159	0.0182	0.224	12.8
<i>s_M</i> :	0.00100	0.0043	0.01190	0.00254	0.00082	0.0181	0.0106	0.044	0.0115	0.087	3.0

The laboratory mean values have been examined statistically to eliminate outlying values (Grubbs test, 99%).

The omission of outlying values is indicated by “---”.

M : Arithmetic mean of the laboratory means

s_M : Standard deviation of the laboratory means

Note: “< - values” were not included into the calculation of *M* and *s_M*.

The line number should not be mistaken for the laboratory code number.

Participating Laboratories

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany)

ESK Ceramics GmbH & Co. KG, Kempten (Germany)

ESK-SiC GmbH, Frechen (Germany)

GrafTech International Holdings Inc., Parma – OH (USA)

H.C. Starck GmbH, Goslar (Germany)

H.C. Starck GmbH, Laufenburg (Germany)

HORIBA Jobin Yvon S.A.S., Longjumeau Cedex (France)

Institut "Jožef Stefan", Ljubljana (Slovenia)

Leibniz-Institut für Kristallzüchtung, Berlin (Germany)

MERSEN US, Bay City - MI, (USA)

Schunk Kohlenstofftechnik GmbH, Heuchelheim (Germany)

SGL CARBON GmbH, Bonn (Germany)

SGL CARBON GmbH, Meitingen (Germany)

SGL Carbon S.A.S., Chedde (France)

South African Nuclear Energy Corporation (Necsa), Pelindaba (South Africa)

Spectro Analytical Instruments, Kleve (Germany)

thyssenkrupp Steel Europe AG, Duisburg (Germany)

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 35 mg.

It is not required to dehydrate the sample before starting measurements.

Transport and Storage

The material should be stored at room temperature (20 °C) in a dust-free and dry environment. Transport under normal ambient conditions.

Analytical Method used for Determination

Parameter	Line no. (see previous Tables)	Method
Al	1 2, 3, 4, 5, 8, 11, 12, 15, 16, 17, 19, 20 6, 7, 9, 10, 13, 18 14	SS-ET AAS ETV-ICP OES ICP OES MF-DCArc-OES
B	1, 3, 4, 6, 7, 8 2 5	ICP OES DCArc-OES MF-DCArc-OES
Ba	2, 6, 7, 9, 11, 13, 14, 15, 16, 18, 22 3, 4, 5, 8, 10, 17, 19 12 20 21	ETV-ICP OES ICP OES MF-DCArc-OES INAA k_0 -INAA
Be	1, 2, 3, 4, 6, 7 5	ETV-ICP OES MF-DCArc-OES
Ca	1, 2, 3, 4, 5, 6, 8, 9, 10, 14, 19, 20 7 11, 12, 13, 15, 16, 17, 18 21	ETV-ICP OES MF-DCArc-OES ICP OES DCArc-OES
Co	1, 2, 3, 5, 6, 7, 14, 24 4 8 9, 10, 11, 12, 15, 16, 18, 19, 20, 22, 23 13 17 21	ICP OES SS-ET AAS INAA ETV-ICP OES ETV-ICP-MS MF-DCArc-OES k_0 -INAA
Cr	1, 2, 3, 10, 11, 12, 13, 16, 18, 23, 24, 25 4, 5, 6, 7, 8, 14, 15, 20, 22 9 17 19 21 26	ETV-ICP OES ICP OES ETV-ICP-MS MF-DCArc-OES INAA SS-ET AAS k_0 -INAA
Cu	1, 5, 14 2, 3, 4, 6, 7, 8, 9, 10, 12, 13, 15 11 16	ICP OES ETV-ICP OES SS-ET AAS ETV-ICP-MS
Fe	1, 2, 6, 9, 10, 20, 22, 23, 24 3, 4, 5, 7, 8, 11, 12, 13, 15, 17, 18, 21 14 16 19 25	ICP OES ETV-ICP OES DCArc-OES MF-DCArc-OES INAA k_0 -INAA
K	1, 2, 8, 14, 17 3, 5, 7, 10, 11, 13, 15, 16, 18 6 9 12 19	ICP OES ETV-ICP OES MF-DCArc-OES SS-ET AAS ETV-ICP-MS k_0 -INAA
Li	1 2, 4, 5, 6, 7, 8, 9, 10 3	ICP OES ETV-ICP OES MF-DCArc-OES
Mg	1, 5, 6, 7, 11, 12, 13, 18, 19, 20, 21 2, 3, 4, 8, 9, 14, 15 10 16 17	ETV-ICP OES ICP OES SS-ET AAS MF-DCArc-OES DCArc-OES

(continued)

Mn	1, 2, 3, 4, 13, 14, 15, 21 5, 6, 7, 8, 9, 10, 11, 12, 16, 17, 19 18 20	ICP OES ETV-ICP OES ETV-ICP-MS MF-DCArc-OES
Mo	1, 2, 3, 4, 7, 9, 11, 16, 17 5, 6, 8, 12, 13, 14 10 15	ETV-ICP OES ICP OES MF-DCArc-OES k_0 -INAA
Na	1, 15 2, 4, 5, 6, 7, 9, 10, 11, 14, 17 3 8 12 13 16	ICP OES ETV-ICP OES SS-ET AAS INAA k_0 -INAA DCArc-OES MF-DCArc-OES
Ni	1, 2, 4, 5, 6, 11, 18, 22, 23 3 7 8, 9, 10, 12, 13, 14, 15, 16, 19, 20, 21, 24 17	ICP OES DCArc-OES MF-DCArc-OES ETV-ICP OES SS-ET AAS
P	1, 2, 3, 4, 5, 6, 7, 9, 10 8	ETV-ICP OES ICP OES
Pb	1, 2, 3, 4, 5, 6, 8, 9, 10, 14 7, 11, 12, 13, 15, 16	ETV-ICP OES ICP OES
S	1, 3, 4, 5, 6, 7, 8, 9, 10 2	ETV-ICP OES ICP OES
Si	2 3 4, 5, 6, 7, 12 8, 9 10, 11	MF-DCArc-OES SS-ET AAS ICP OES DCArc-OES Photometry
Sr	1, 3, 4, 6, 8, 9, 11, 14, 16 2, 5, 7, 12, 15 10 13	ETV-ICP OES ICP OES MF-DCArc-OES ETV-ICP-MS
Ti	1, 13, 17, 18, 19, 20, 21, 22 2, 3, 4, 5, 6, 8, 10, 11, 12, 14, 15, 16 7 9	ICP OES ETV-ICP OES DCArc-OES MF-DCArc-OES
V	1, 4, 5, 7, 9, 11, 12, 16, 17, 19 2, 3, 6, 8, 10, 13, 14, 15, 18 20	ETV-ICP OES ICP OES MF-DCArc-OES
W	1, 9, 10, 11, 12, 15, 16, 17, 18 2, 3, 4, 5, 6, 7, 8, 13 14	ETV-ICP OES ICP OES k_0 -INAA
Y	1, 2, 3, 4, 5, 6, 7, 8, 10, 11 9	ETV-ICP OES ETV-ICP-MS
Zn	1, 2, 3, 5, 6, 8, 9, 10, 11, 12, 14 4, 13, 16, 18 7 15 17	ETV-ICP OES ICP OES INAA k_0 -INAA ETV-ICP-MS
Zr	1, 5, 12, 15, 18, 19, 20, 21 2, 3, 4, 6, 7, 8, 9, 10, 11, 13, 14, 22 16 17 23	ICP OES ETV-ICP OES MF-DCArc-OES DCArc-OES k_0 -INAA
Ag	1 2 3, 4, 5, 8 6, 10 7 9	SS-ET AAS MF-DCArc-OES ETV-ICP OES ICP OES INAA k_0 -INAA

(continued)

<i>As</i>	1, 3, 4, 5 2 6	<i>ETV-ICP OES</i> <i>INAA</i> <i>ETV-ICP-MS</i>
<i>Au</i>	1 2, 3	<i>k₀-INAA</i> <i>ICP OES</i>
<i>B_{ETV}</i>	1, 2, 3, 4, 5, 6, 7, 8	<i>ETV-ICP OES</i>
<i>Bi</i>	1 2, 3, 4 5	<i>ETV-ICP-MS</i> <i>ETV-ICP OES</i> <i>ICP OES</i>
<i>Cd</i>	1 2, 3, 4, 5, 7, 9, 10 8 6, 11, 13, 14	<i>SS-ET AAS</i> <i>ETV-ICP OES</i> <i>ETV-ICP-MS</i> <i>ICP OES</i>
<i>Cs</i>	1 2	<i>k₀-INAA</i> <i>INAA</i>
<i>Dy</i>	1, 2, 3	<i>ICP OES</i>
<i>Eu</i>	1, 4 2 3	<i>ICP OES</i> <i>INAA</i> <i>k₀-INAA</i>
<i>Ga</i>	1	<i>ETV-ICP OES</i>
<i>Gd</i>	1, 2, 3	<i>ICP OES</i>
<i>Hg</i>	1 2 3, 4	<i>k₀-INAA</i> <i>SS-ET AAS (DMA-80)</i> <i>ICP OES</i>
<i>In</i>	1, 2	<i>ICP OES</i>
<i>La</i>	1	<i>INAA</i>
<i>Nb</i>	1	<i>ETV-ICP OES</i>
<i>Rb</i>	1	<i>INAA</i>
<i>Re</i>	1 2, 3	<i>k₀-INAA</i> <i>ICP OES</i>
<i>Rh</i>	1, 2	<i>ICP OES</i>
<i>Sb</i>	1 2 3 4, 7, 11, 12 5, 6, 8, 9, 10	<i>INAA</i> <i>k₀-INAA</i> <i>SS-ET AAS</i> <i>ICP OES</i> <i>ETV-ICP OES</i>
<i>Sc</i>	1 2 3 4 5	<i>ETV-ICP OES</i> <i>ICP OES</i> <i>INAA</i> <i>k₀-INAA</i> <i>ETV-ICP-MS</i>
<i>Se</i>	1	<i>INAA</i>
<i>Si_{ETV}</i>	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	<i>ETV-ICP OES</i>
<i>Sm</i>	1, 3 2	<i>ICP OES</i> <i>k₀-INAA</i>
<i>Sn</i>	1, 3, 4, 6, 7 2 5	<i>ETV-ICP OES</i> <i>ICP OES</i> <i>MF-DCArc-OES</i>
<i>Ta</i>	1 2, 4 3	<i>k₀-INAA</i> <i>ICP OES</i> <i>ETV-ICP OES</i>
<i>Tb</i>	1 2	<i>ICP OES</i> <i>k₀-INAA</i>
<i>Te</i>	1, 2	<i>ETV-ICP OES</i>
<i>Th</i>	1 2	<i>ICP OES</i> <i>k₀-INAA</i>
<i>U</i>	1 2, 4 3	<i>k₀-INAA</i> <i>ICP OES</i> <i>ETV-ICP OES</i>
<i>Ash</i>	1	<i>DIN 51903 (Gravimetry)</i>

Entries emphasized in *italics* refer to values that have been assigned "for information" only.

Abbreviations:

DCArc-OES	Direct current arc optical emission spectrometry
ETV-ICP OES	Inductively coupled plasma optical emission spectrometry with electrothermal vaporisation
ETV-ICP-MS	Inductively coupled plasma mass spectrometry with electrothermal vaporisation
ICP OES	Inductively coupled plasma optical emission spectrometry
INAA	Instrumental neutron activation analysis
k_0 -INAA	k_0 -Instrumental neutron activation analysis
MF-DCArc-OES	Magnetically stabilised direct current arc optical emission spectrometry
SS-ET AAS	Solid sampling electrothermal atomic absorption spectrometry
DMA-80	Direct mercury analyser (Thermal combustion, reduction, amalgamation and detection by AAS)

Metrological Traceability

The values are traceable to the SI (Système International d'Unités) via calibration using pure metals or substances of known stoichiometry.

Safety Information

The usual laboratory safety precautions have to be applied. Graphite powder is not known to be toxic. No hazardous effect is to be expected if the material is used under conditions usually adopted in analytical laboratories when handling finely dispersed powder materials. It is strongly recommended to handle and dispose the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

Technical Report

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-S009 is available on request or can be downloaded from BAM website (www.bam.de)

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