

Bundesanstalt für Materialforschung und -prüfung (BAM)

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

## Certified Reference Material

### BAM-S010

High Purity Graphite Powder

#### Certified Values

Parameter	Mass fraction <sup>1)</sup> in mg/kg	Uncertainty <sup>2)</sup> in mg/kg
Al	0.047	0.011
Ba	0.0023	0.0009
Be	0.000016	0.000008
Ca	0.061	0.029
Co	0.0008	0.0004
Cr	0.011	0.005
Cu	0.0049	0.0020
Fe	0.21	0.06
K	0.021	0.009
Li	0.00017	0.00010
Mg	0.019	0.005
Mn	0.0029	0.0008
Na	0.037	0.011
Ni	0.087	0.024
P	0.027	0.011
Pb	0.0029	0.0014
S	5.7	0.5
Sr	0.0008	0.0004
Ti	0.011	0.008
W	0.0036	0.0011
Y	0.00019	0.00010
Zn	0.014	0.008
Zr	0.0016	0.0005

<sup>1)</sup> 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.

<sup>2)</sup> 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 there is a revocation from the producer of the material.

## Values for information

Parameter	Mass fraction <sup>1)</sup> in mg/kg	Uncertainty <sup>2)</sup> in mg/kg
Ag	< 0.001	
As	0.0034	0.0008
B <sup>3)</sup>	0.0040	0.0019
Cd	0.00016	0.00009
Mo	0.0040	0.0019
Sb	< 0.03	
Si <sup>3)</sup>	0.27	0.09
Sn	< 0.02	
Te	< 0.002	
V	0.005	0.003

<sup>1)</sup> 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.

<sup>2)</sup> 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).

<sup>3)</sup> 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). Using ETV-ICP-OES for determination, lower mass fractions could be obtained than with other methods. In case of BAM-S010 the differences could not be observed because there was no dataset for boron and only one data set for Si obtained with other methods than ETV-ICP-OES. Therefore, the mass fractions of B and Si are only given for information.

### Further informative values (laboratory mean values without statistical evaluation)

Line no.	Mass fraction in mg/kg							
	<i>Au</i>	<i>Bi</i>	<i>Cs</i>	<i>Eu</i>	<i>Hg</i>	<i>La</i>	<i>Rb</i>	<i>Re</i>
1	<i>&lt; 0.00003</i>	<i>0.0013</i>	<i>&lt; 0.0004</i>	<i>&lt; 0.00017</i>	<i>0.0049</i>	<i>0.0031</i>	<i>&lt; 0.006</i>	<i>0.00051</i>
2		<i>0.0023</i>	<i>&lt; 0.002</i>	<i>&lt; 0.0003</i>	<i>0.0071</i>			
(continued)								
Line no.	<i>Sc</i>	<i>Se</i>	<i>Sm</i>	<i>Ta</i>	<i>Tb</i>	<i>Th</i>	<i>U</i>	
1	<i>0.00004</i>	<i>&lt; 0.0042</i>	<i>&lt; 0.00005</i>	<i>0.00054</i>	<i>&lt; 0.0008</i>	<i>&lt; 0.0011</i>	<i>&lt; 0.0008</i>	
2	<i>&lt; 0.00018</i>			<i>&lt; 0.0009</i>				
3	<i>0.00021</i>							

## Sample Description

The reference material BAM-S010 consists of a high 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. It is not suitable for the production of pressed pellets without additives.

## Participating Laboratories

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany)  
 3M Technical Ceramics ZNL der 3M Deutschland GmbH, Kempten (Germany)  
 ESK-SiC GmbH, Frechen (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)  
 Spectro Analytical Instruments, Kleve (Germany)

## Means of Accepted Data Sets

Certified values (mass fraction in mg/kg)

Line no.	Al	Ba	Be	Ca	Co	Cr	Cu	Fe	K	Li	Mg	Mn
1	0.0328	0.0012	0.000011	0.0282	0.00033	0.0058	0.00151	0.070	0.0084	0.000041	0.0086	0.00154
2	0.0368	0.0013	0.000012	0.0369	0.00038	0.0063	0.00276	0.168	0.0100	0.000057	0.0123	0.00207
3	0.0368	< 0.002	0.000013	0.0408	0.00052	0.0064	0.00333	0.173	0.0111	0.000082	0.0150	0.00262
4	0.0410	0.0020	0.000015	0.0439	0.00059	0.0070	0.00345	0.192	0.0155	0.000130	0.0152	0.00268
5	0.0414	0.0022	0.000029	0.0511	0.00065	0.0077	0.00392	0.197	0.0183	0.000150	0.0161	0.00278
6	0.0420	0.0023	---	0.0542	0.00070	0.0084	0.00428	0.200	0.0194	0.000175	0.0182	0.00283
7	0.0432	0.0024	---	0.0665	0.00077	0.0085	0.00440	0.202	< 0.02	0.000363	0.0205	< 0.003
8	0.0499	0.0027		0.0684	< 0.0015	0.0092	0.00750	0.212	< 0.24	0.000384	0.0209	0.00332
9	0.0517	0.0028		0.0688	0.00176	0.0095	0.00758	0.223	0.0292	---	0.0218	0.00338
10	0.0534	0.0034		0.0794	0.00188	0.0114	< 0.01	0.265	0.0323	< 0.005	0.0220	0.00395
11	0.0583	---		0.0845	< 0.002	0.0144	0.01028	0.292			0.0225	0.00422
12	0.0606	< 0.27		0.1042	< 0.004	0.0177		0.295			0.0234	---
13	0.0606			---	< 0.005	0.0252		---			0.0246	
14					---	---		---				
<i>M</i> :	0.0468	0.0023	0.000016	0.0606	0.00084	0.0106	0.00490	0.207	0.0209	0.000173	0.0185	0.00294
<i>S<sub>M</sub></i> :	0.0095	0.0007	0.000008	0.0222	0.00058	0.0056	0.00269	0.061	0.0120	0.000132	0.0048	0.00081

Line no.	Na	Ni	P	Pb	S	Sr	Ti	W	Y	Zn	Zr
1	0.017	0.0668	0.0125	0.00116	---	0.00045	0.0044	0.00177	0.000075	0.0059	0.00057
2	0.020	0.0690	0.0168	0.00129	4.73	0.00060	< 0.005	0.00261	0.000103	0.0073	< 0.001
3	0.026	0.0763	0.0201	0.00145	5.37	0.00062	0.0051	0.00263	0.000145	0.0076	0.00113
4	0.026	0.0773	0.0342	0.00176	5.38	0.00069	0.0059	0.00307	0.000197	0.0081	0.00149
5	0.028	0.0778	0.0357	< 0.0025	5.77	0.00082	0.0074	0.00433	0.000289	0.0090	0.00151
6	0.030	0.0789	0.0396	0.00266	5.93	0.00148	0.0090	0.00447	0.000342	0.0097	0.00187
7	0.037	0.0802	---	0.00359	6.14	< 0.002	0.0095	0.00477	< 0.001	0.0105	0.00198
8	0.037	0.0815		0.00505	6.15	---	0.0098	0.00487		0.0109	0.00202
9	0.037	0.0855		0.00586	6.28		0.0233	< 0.1		0.0112	0.00226
10	0.060	0.0902		---			0.0257			0.0160	< 0.004
11	0.060	0.1055		< 0.1						0.0216	
12	0.068	0.1141								0.0280	
13	---	0.1274								0.0346	
14										---	
15										< 0.08	
<i>M</i> :	0.037	0.0870	0.0265	0.00285	5.72	0.00078	0.0111	0.00356	0.000192	0.0139	0.00160
<i>S<sub>M</sub></i> :	0.017	0.0180	0.0114	0.00181	0.53	0.00037	0.0079	0.00119	0.000106	0.0089	0.00055

Informative values (mass fraction in mg/kg)

Line no.	Ag	As	B	Cd	Mo	Sb	Si	Sn	Te	V
1	0.000058	0.00245	0.00117	0.000071	0.00248	0.0011	0.155	0.00162	0.00028	0.0020
2	0.000159	0.00350	0.00171	0.000072	0.00286	0.0020	0.167	0.00423	0.00095	0.0029
3	0.000296	0.00377	0.00227	0.000103	0.00370	0.0024	0.180	0.01785	0.00193	0.0033
4	<0.0013	0.00386	0.00388	0.000122	0.00386	0.0061	0.202			0.0041
5	<0.012	---	0.00612	< 0.00013	0.00422	0.0065	0.216			0.0049
6		<0.1	0.00614	0.000270	0.00491	0.0065	0.249			0.0049
7			0.00635	0.000300	0.00509	0.0203	0.250			< 0.005
8			<0.02	---	0.00518	<0.1	0.306			0.0050
9					< 0.006		0.341			0.0067
10					<0.1		0.440			0.0096
11							0.443			---
<i>M</i> :	0.000171	0.00339	0.00395	0.000156	0.00404	0.0064	0.268	0.00790	0.00105	0.0048
<i>s<sub>M</sub></i> :	0.000119	0.00065	0.00227	0.000102	0.00101	0.0066	0.101	0.00872	0.00083	0.0023

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

Note: "< - values" were not included into the calculation of *M* and *s<sub>M</sub>*.

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

## 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 30 seconds 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, 4 - 13	ETV-ICP-OES
	3	SS-ET AAS
Ba	1 - 10	ETV-ICP-OES
	12	k <sub>0</sub> -INAA
Be	1 - 5	ETV-ICP-OES
Ca	1, 3 - 12	ETV-ICP-OES
	2	SS-ET AAS
Co	1 - 7, 9, 10, 11, 13	ETV-ICP-OES
	8	SS-ET AAS
	12	k <sub>0</sub> -INAA
Cr	1 - 9, 11	ETV-ICP-OES
	10	SS-ET AAS
	12	INAA
	13	k <sub>0</sub> -INAA
Cu	1	SS-ET AAS
	2 - 11	ETV-ICP-OES
Fe	1	SS-ET AAS
	2-12	ETV-ICP-OES
K	1, 3 - 7, 9, 10	ETV-ICP-OES
	2	SS-ET AAS
	8	k <sub>0</sub> -INAA
Li	1 - 8, 10	ETV-ICP-OES

<b>Parameter</b>	<b>Line no. (see previous Tables)</b>	<b>Method</b>
Mg	1, 3 - 13 2	ETV-ICP-OES SS-ET AAS
Mn	1 - 7, 9 - 11 8	ETV-ICP-OES SS-ET AAS
Na	1 - 3, 5 - 8, 10 - 12 4 9	ETV-ICP-OES SS-ET AAS k <sub>0</sub> -INAA
Ni	1 - 11, 13 12	ETV-ICP-OES SS-ET AAS
P	1 - 6	ETV-ICP-OES
Pb	1 - 4, 6 - 9, 11 5	ETV-ICP-OES SS-ET AAS
S	2 - 9	ETV-ICP-OES
Sr	1 - 7	ETV-ICP-OES
Ti	1 - 10	ETV-ICP-OES
W	1 2 - 9	k <sub>0</sub> -INAA ETV-ICP-OES
Y	1 - 7	ETV-ICP-OES
Zn	1 - 8, 10 - 12 9 13 15	ETV-ICP-OES SS-ET AAS INAA k <sub>0</sub> -INAA
Zr	1 - 10	ETV-ICP-OES
Ag	1 - 3 4 5	<i>ETV-ICP-OES</i> <i>INAA</i> <i>k<sub>0</sub>-INAA</i>
As	1, 3, 4, 6 2	<i>ETV-ICP-OES</i> <i>INAA</i>
Au	1	<i>k<sub>0</sub>-INAA</i>
B	1 - 8	<i>ETV-ICP-OES</i>
Bi	1, 2	<i>ETV-ICP-OES</i>
Cd	1 - 4, 6, 7 5	<i>ETV-ICP-OES</i> <i>SS-ET AAS</i>
Cs	1 2	<i>INAA</i> <i>k<sub>0</sub>-INAA</i>
Eu	1, 2	<i>k<sub>0</sub>-INAA</i>
Hg	1 2	<i>k<sub>0</sub>-INAA</i> <i>SS-ET AAS (DMA-80)</i>
La	1	<i>INAA</i>
Mo	1 - 8, 10 9	<i>ETV-ICP-OES</i> <i>k<sub>0</sub>-INAA</i>
Rb	1	<i>INAA</i>
Re	1	<i>k<sub>0</sub>-INAA</i>
Sb	1 2 3 - 8	<i>k<sub>0</sub>-INAA</i> <i>INAA</i> <i>ETV-ICP-OES</i>
Sc	1 2 3	<i>ETV-ICP-OES</i> <i>k<sub>0</sub>-INAA</i> <i>INAA</i>
Se	1	<i>INAA</i>
Si	1 2 - 11	<i>SS-ET AAS</i> <i>ETV-ICP-OES</i>
Sm	1	<i>k<sub>0</sub>-INAA</i>
Sn	1 - 3	<i>ETV-ICP-OES</i>
Ta	1 2	<i>ETV-ICP-OES</i> <i>k<sub>0</sub>-INAA</i>
Tb	1	<i>k<sub>0</sub>-INAA</i>
Te	1 - 3	<i>ETV-ICP-OES</i>
Th	1	<i>k<sub>0</sub>-INAA</i>
U	1	<i>k<sub>0</sub>-INAA</i>
V	1 - 10	<i>ETV-ICP-OES</i>

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

## Abbreviations:

ETV-ICP-OES	Inductively coupled plasma optical emission spectrometry with electrothermal vaporisation
INAA	Instrumental neutron activation analysis
$k_0$ -INAA	$k_0$ -Instrumental neutron activation analysis
SS-ET AAS	Solid sampling electrothermal atomic absorption spectrometry
DMA-80	Direct mercury analyser (thermal combustion, reduction, amalgamation and detection by AAS)

## Metrological Traceability

To ensure traceability of the certified mass fractions to the SI (Système International d'Unités) calibration was performed using standard solutions prepared from pure metals or stoichiometric compounds or with traceable commercial calibration solutions.

## 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-S010 is available from BAM website ([www.bam.de](http://www.bam.de))

**Accepted as BAM-CRM on 2024-01-16**

**Bundesanstalt für Materialforschung und -prüfung (BAM)**

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