MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 360

SIALON BONDED SILICON CARBIDE

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, FRANCE, GERMANY, and LUXEMBOURG, issued by the Bureau of Analysed Samples Ltd.

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

1 OLIVER, G. J., *BSc, PhD, CChem, MRSC, MICeram,* British Ceramic Research Ltd., Stoke-on-Trent.

2 PAGE-GIBSON, J. E., *BSc, CChem, MRSC,* Ridsdale and Co. Ltd., Middlesbrough.

ANALYSTS representing MANUFACTURERS and USERS

3 CRAWSHAW, J., *LRSC,* Dyson Industries Ltd., Sheffield.

4 HASSLER, J., Electroschmelzwerk Kempten GmbH., Kempten, Germany.

ANALYSTS representing MANUFACTURERS and USERS (cont.)

5 KIRKHAM, R., Hepworth Refractories Ltd., Worksop.

6 LAURENT, Y., Luxcontrol S.A., Esch-sur-Alzette, Luxembourg.

7 RAVAINE, D., Institut de Recherches de la Sidérurgie Francaise (IRSID), Maizières-lès-Metz, France.

8 REEVE, Mrs. B.C.E., *LRSC* Morgan Material Technology Ltd., Stourport-on-Severn.

ANALYSES

Mean of 4 values - mass content in %. All results relate to the dried (105ºC) sample.

Analyst No.	Total C	Total Si	Total Al	Ca	Ti	*Free C*	*Free Si*	*Total Fe*	*O*	*N*
1	...	60.5200	6.5515	0.1163	0.0203	*0.0775*	...	*0.1623*	...	...
2	23.5286	60.5220	6.4086	0.1156	0.0283	*0.1092*	*0.4608*	*0.2251*	...	...
3	...	60.1895	6.5594	0.1245	0.0273	*0.0775*	...	*0.1813*	...	...
4	23.5725	61.1050	6.5350	0.1020	0.0179	*0.0810*	*0.8325*	*0.1488*	*3.8998*	*4.7550*
5	...	61.1730	6.4570	0.1232	0.0279	*0.0650*	...	*0.1643*	*4.1475*	*4.8425*
6	23.4900	60.6500	6.5800	0.1225	0.0175	*0.1400*	...	...	...	...
7	23.8028	61.3000	6.5125	0.1288	0.0320	*0.0465*	*0.5470*	*0.2325*	*3.9625*	*4.6975*
8	23.2500	60.9250	6.5600	0.0900	0.0300	...	*0.3100*	*0.2200*	*4.1150*	*4.7750*
M_M	**23.5288**	**60.7931**	**6.5205**	**0.1154**	**0.0252**	***0.085***	***0.538***	***0.1906***	***4.03***	***4.77***
s_M	0.1976	0.3795	0.0590	0.0131	0.0057	...	...	...	...	...
s_W	0.1411	0.1850	0.1028	0.0061	0.0036	...	...	...	...	...

The above figures are those which each Analyst has decided upon after careful verification.
Figures in bold type certified, figures in small italic type only approximate.

M_M: Mean of the laboratory mean values. s_M: standard deviation of the laboratory mean values. s_W: Intralaboratory standard deviation.

The following additional information was supplied by two or more Analysts: Cr <0.01%, Mg <0.02%, Mn <0.01%, Na <0.01%, Ni 0.013%, K <0.01%

CERTIFIED VALUES (Cv)

mass content in %

	Total C	Total Si	Total Al	Ca	Ti
Cv	**23.53**	**60.8**	**6.52**	**0.115**	**0.025**
C(95%)	0.25	0.3	0.05	0.011	0.005

The half width confidence interval, **C**(95%), is an expression of the uncertainty of the certified value.

$\mathbf{C}(95\%) = \frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate two sided Student's t value at the 95% confidence level for "n" acceptable mean values.

For further information regarding the confidence interval for the certified value see ISO Guide 35.

NB: Although widely accepted within the industry "mass content in %" is neither an SI nor an IUPAC supported quantity.

Multiplication of the certified value (Cv) by 10^4 will yield the value in µg/g.

Date of this edition: October 2018.

(The current edition is available at www.basrid.co.uk)

BCS-CRM 360 SIALON BONDED SILICON CARBIDE

NOTES ON METHODS USED

TOTAL CARBON

Analysts Nos. 2, 4 & 8 determined total carbon gravimetrically following combustion at 1050°C using a lead borate flux (DIN 51075 part 3). Nos 6 and 7 determined carbon after high frequency combustion, No. 6 by coulometry and No. 7 by infrared absorption.

TOTAL SILICON

Analysts Nos. 1, 3, 5, 6, 7 and 8 determined total silicon by X-Ray Fluorescence Spectrometry - Fused Bead Technique. No. 2 determined total silicon gravimetrically after dehydration with perchloric acid after fusion with sodium carbonate/peroxide. No. 4 used Inductively Coupled Plasma-Optical Emission Spectrometry after fusion with sodium carbonate/boric acid.

TOTAL ALUMINIUM

Analysts Nos. 1, 3, 5 6 and 7 determined total aluminium by X-Ray Fluorescence Spectrometry - Fused Bead Technique. Nos. 2 and 8 used Flame Atomic Absorption Spectrometry after fusion with lithium metaborate and vanadium pentoxide. No. 4 used Inductively Coupled Plasma-Optical Emission Spectrometry after fusion with sodium carbonate/boric acid.

CALCIUM

Analysts Nos. 1, 3, 5, 6 and 7 determined calcium by X-Ray Fluorescence Spectrometry - Fused Bead Technique. Nos. 2 and 8 used Flame Atomic Absorption Spectrometry and No. 4 used Inductively Coupled Plasma-Optical Emission Spectrometry.

TITANIUM

Analysts Nos. 1, 3, 5, 6 and 7 determined titanium by X-Ray Fluorescence Spectrometry - Fused Bead Technique. Nos. 2 and 8 determined titanium photometrically, No. 2 with diantipyrylmethane. No. 4 used Inductively Coupled Plasma-Optical Emission Spectrometry.

FREE CARBON

Analysts Nos. 1, 3 and 5 determined free carbon by the loss on ignition at 700-750°C. No. 2 determined free carbon gravimetrically after combustion at 750°C (DIN51075 part 2). No. 4 used a similar method. Nos. 6 and 7 determined free carbon coulometrically.

FREE SILICON

Analyst No. 2 determined free silicon by silver displacement from AgF after prior treatment with an acid wash to remove free iron (AFNOR NF B49-421). Nos. 4, 7 and 8 determined free silicon by hydrogen evolution (DIN 51075 part 4).

TOTAL IRON

Analysts Nos. 1, 3, 5 and 7 determined total iron by X-Ray Fluorescence Spectrometry - Fused Bead Technique. No. 2 used Flame Atomic Absorption Spectrometry. No. 4 used Inductively Coupled Plasma-Optical Emission Spectrometry after decomposition of the sample with $HF/HNO_3/H_2SO_4$ in a high pressure digestion vessel. No. 8 determined iron photometrically.

OXYGEN

All Analysts determined oxygen by infrared absorption after fusion.

NITROGEN

Analyst No. 4 determined nitrogen after conversion to ammonia with LiOH. Nos. 5, 7 and 8 by thermal conductivity after fusion.

DESCRIPTION OF SAMPLE

Bottles of 100g of powder crushed to pass through a 125 micron (120 mesh) sieve from which the fines passing through a 53 micron (300 mesh) sieve have been rejected. The sieved material was passed over a magnetic separator.

The preparation of representative samples for chemical analysis and the certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

INTENDED USE

This sample is intended for uses such as the verification of the accuracy and repeatability of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments, for establishing values for secondary reference materials and for training purposes.

The sample should be mixed thoroughly before each use.

STABILITY

BCS-CRM 360 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. When the bottle has been opened the lid should be secured immediately after use.

TRACEABILITY

The characterisation of this material has been achieved by chemical analysis involving inter-laboratory study, each laboratory using the method of their choice, details of which are given above.

Most of the analytical methods used in the characterisation of this CRM were either international or national standard methods or methods which are technically equivalent. All laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 360 has been established by multiplying the standard error arising from the chemical analysis by the appropriate two sided Student's t value at the 95% confidence level for the number of results.

COMMUTABILITY

BCS-CRM 360 is intended to be used in the same physical form as that used by the participating Analysts and therefore commutability is not of relevance in respect of this CRM.

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by Bureau of Analysed Samples Ltd may be obtained from the address below.

NEWHAM HALL, NEWBY,
MIDDLESBROUGH, ENGLAND, TS8 9EA
Email: enquiries@basrid.co.uk
Website: www.basrid.co.uk

For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
Managing Director

Preliminary Edition ..... February 1995
Main Edition ..... June 1996
Main Edition (revised with editorial changes) ..... October 2018