BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

4004

CERTIFICATE OF ANALYSIS

BCS-CRM No. 305/2

FERRO-SILICON

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, ESTONIA, HUNGARY and THE NETHERLANDS, issued by the Bureau of Analysed Samples Ltd.

ANALYSES

Mean of 4 values - mass content in %

Analyst	C	Si	Mn	P	Al	Ca	*S*	*Cr*	*Ti*	*Mg*	*Fe*
1	0.1162	77.044	0.1533	0.0300	1.1127	0.2707	...	...	...	...	...
2	…	76.595	0.1474	0.0296	1.1386	0.2598	...	...	...	...	...
3	…	77.030	0.1600	0.0256	1.0975	0.2775	...	...	...	...	...
4	0.0987	76.953	0.1435	0.0305	1.1063	0.2671	...	...	...	...	...
5	0.1108	76.913	0.1478	0.0285	1.1225	0.2575	...	...	...	...	...
6	…	76.443	0.1620	0.0328	1.1670	0.2943	...	...	...	...	...
7	0.1138	…	…	…	…	…	...	...	...	...	...
8	0.1199	76.640	0.1382	0.0243	1.0628	0.2611	*0.001*	*0.07*	*0.08*	*0.03*	*20.1*
9	0.1212	…	0.1623	0.0325	1.1881	0.2681	*0.002*	*0.09*	*0.08*	*0.04*	*19.9*
M_M	**0.1134**	**76.802**	**0.1518**	**0.0292**	**1.1244**	**0.2695**					
s_M	0.0082	0.240	0.0091	0.0031	0.0398	0.0120					
s_W	0.0039	0.075	0.0040	0.0006	0.0101	0.0073					

M_M: Mean of the laboratory mean values. **s_M**: standard deviation of the laboratory mean values. **s_W**: average within laboratory standard deviation.

Analysts Nos. 2 and 5 also provided information values for Si, No. 2 reporting 75.89% by ICP-OES and No. 5 giving 77.16% by XRF.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	Al	Ca
Cv	**0.113**	**76.80**	**0.152**	**0.029**	**1.12**	**0.270**
C(95%)	0.009	0.23	0.008	0.003	0.04	0.010
Minimum weight (g)	0.15	0.2	0.1	0.1	0.1	0.1

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

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.

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

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NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 5 and 7 determined carbon using combustion/infra-red methods calibrated with carefully selected CRMs (see below for details). Analyst No.4 also used combustion/infra-red, in accordance with ISO 9556 and calibrated with Na_2CO_3. Analysts No. 8 and 9 determined carbon after combustion in a stream of oxygen, No. 8 finishing gravimetrically and No. 9 using a non-aqueous titration.

SILICON

Analysts Nos. 1 and 3 determined silicon using X-Ray Fluorescence Spectrometry (XRF) on a fused bead and calibrating with pure chemicals. The remining analysts used gravimetric methods, mostly dehydrating with perchloric acid, Nos. 2, 4 and 5 following ISO 4158: 1978. Analyst No. 8 used sulphuric acid to dehydrate the silica.

MANGANESE

Analysts Nos. 1, 2, 3, 4 and 6 used Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) to determine manganese, No. 4 using a method based on ISO 4139. Analyst No. 5 used XRF on a fused bead, after calibration with pure chemicals, No.8 used Flame Atomic Absorption Spectrometry (FAAS) and No. 9 determined manganese photometrically after oxidation with periodate.

PHOSPHORUS

Analysts Nos. 1, 2 and 6 determined phosphorus by ICP-OES. Analysts Nos. 3 and 5 used XRF, on a fused bead with pure chemical calibration. Analysts Nos. 4, 8 and 9 used photometric methods, No. 4 as molybdenum blue, as per ASTM E1070, whilst the others extracted the yellow phosphovanadomolybdate complex into 4-Methylpentan-2-one.

ALUMINIUM

Analysts Nos. 1, 2, 6 and 9 determined aluminium by ICP-OES. Nos. 3 and 5 used XRF, on a fused bead with pure chemical calibration, whilst Nos. 4 and 8 used FAAS, No. 4 following ISO 4139:1979.

CALCIUM

With the exception of Analysts Nos. 3 and 5, who used XRF, and No. 8 who used FAAS, all of the Analysts determined calcium by ICP-OES, No. 4 using a method based on ISO 4139.

SULPHUR

Analyst No. 8 determined sulphur by acid-base titration following combustion in oxygen, whilst No. 9 used ICP-OES.

CHROMIUM

Analyst No. 8 determined chromium using FAAS and No. 9 used ICP-OES.

TITANIUM

Both Analysts determined titanium by ICP-OES.

MAGNESIUM

Analyst No. 8 determined magnesium by FAAS whereas No 9 used ICP-OES

IRON

Both Analysts determined iron by titration with Cr(VI) after reduction with Sn(II).

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CO-OPERATING ANALYSTS

1	BLUNDELL, P.,	ALS Inspection UK, Prescot.
2	KONDOROSI, G.,	Dunaferr Labor Nonprofit Kft, Dunaújváros, Hungary.
3	DERUITER, G.,	A H Knight International Ltd., St Helens.
4	KRASNOPEVTSEV, G.,	Bureau Veritas, Harjumaa, Estonia.
5	CULLEY, N.,	Laboratorium voor Multielement Analyses (LMA), Raamsdonksveer, The Netherlands.
6	ABELL, R.,	Laboratory Services International (LSI), Rotterdam, The Netherlands.
7	CLARK, J.,	NSG European Technical Centre, Lathom.
8	ATKINSON, M. J.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
9	JONES, S. J.,	Ridsdale & Co Ltd., Middlesbrough.

DESCRIPTION OF SAMPLE

British Chemical Standard BCS-CRM 305/2 is sold in the form of a powder passing a nominal 150µm aperture sieve. It is supplied in bottles containing 100g.

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

Bureau of Analysed Samples Ltd is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS-CRM 305/2 as defined in BS EN ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with BS EN ISO 17034 and ISO Guide 35.

INTENDED USE

BCS-CRM 305/2 is intended for the verification 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.

In order to ensure that a fully representative sample is taken users should take not less than the minimum weight stated on the certificate for that element, this being the lowest sample mass used by any of the Co-operating Analysts contributing to the certified value. Users of this material should be aware that the use of a smaller sub-sample size may invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use.

STABILITY

BCS-CRM 305/2 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. Once 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. With the sole exception of carbon 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.

For carbon half of the results were also obtained using stoichiometric analytical techniques and methods which were calibrated against pure stoichiometric compounds. However, Analysts Nos. 4, 5 and 7 used combustion infrared methods which had been calibrated using five carefully selected certified reference materials. The preferred option would be to select ferro-silicon CRMs similar in composition to BCS-CRM 305/2, but, in this case, there were insufficient CRMs of appropriate quality to allow this approach. However, both theoretical considerations and practical experience have demonstrated that unalloyed steels can be used successfully to calibrate combustion/infrared instruments to determine carbon in ferro-silicon and this approach has therefore been adopted here, using CRMs which demonstrate full unbroken traceability to the SI. Different Analysts used different combinations of CRMs, but the CRMs used were as follows: BCS-CRMs 111, 456/2 and 458/2, ECRMs 057-2, 086-1, 087-1, 096-2 and the ferro-silicon JK 39.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 305/2 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. Homogeneity has been assessed in accordance with ASTM E826 – 85 and found to be acceptable. Homogeneity has not, therefore, been included in the calculated measurement uncertainty. The stability of this CRM and its transportation also make negligible contributions to the overall uncertainty of the certified values.

COMMUTABILTY

BCS-CRM 305/2 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.

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For BUREAU OF ANALYSED SAMPLES LTD

R P MEERES,
Managing Director
September 2020