



CERTIFICATE OF ANALYSIS
BCS-CRM No. 535
HIGH CARBON IRON POWDER

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, AUSTRIA, CHINA, CZECH REPUBLIC, FINLAND, HUNGARY, INDIA, POLAND, The NETHERLANDS, REPUBLIC OF NORTH MACEDONIA, SPAIN, SWEDEN, and The UNITED STATES OF AMERICA
issued by the Bureau of Analysed Samples Ltd.

ANALYSES

Mean of 4 values - mass content in %.

Analyst	C	Si	Mn	P	S	Cr	Mo	Ni	Al	As	Cu
1	3.874	...	0.471	...	0.0254	0.0498	0.0030	1.377	0.1360
2	0.463	0.1519	0.0277	0.0555	0.0031	...	0.0075	0.0057	...
3	3.861	2.645	0.470	0.1405	0.0293	0.0483	0.0027	...	0.0087	0.0065	0.1413
4	3.860	2.706	0.440	0.1498	0.0295	0.0490	0.0048	1.355	0.0072	0.0056	0.1399
5	...	2.771	0.456	0.1415	...	0.0500	0.0087	0.0054	0.1351
6	0.442	0.1465	...	0.0545	0.0038	...	0.0097	0.0049	...
7	3.831	2.759	0.456	0.1511	0.0281	0.0496	...	1.332	0.0074	0.0062	...
8	3.880	2.713	0.480	0.1500	0.0291	...	0.0035	1.358	0.0073	0.0030	...
9	...	2.734	0.476	0.1431	...	0.0510	0.0029	1.363	...	0.0064	0.1432
10	...	2.676	0.454	0.1485	...	0.0527	0.0031	1.317	0.0074	0.0058	0.1345
11	3.905	2.699	0.458	...	0.0294	0.0534	0.0042	...	0.0106	0.0052	0.1404
12	...	2.659	0.449	0.1358	...	0.0495	0.0039	1.345	0.0102	0.0041	0.1458
13	3.865	2.741	0.466	0.1489	0.0293	0.0459	...	1.338	0.1371
14	...	2.693	0.464	0.0498	...	1.357	0.1364
15	3.888	...	0.445	0.1396	0.0288	0.0484	0.0033	1.309	0.0083	0.0037	0.1445
16	3.897	2.684	0.458	0.1357	0.0277	0.0499	...	1.362	0.0079	0.0030	0.1383
17	3.888	2.705	0.462	0.1526	0.0253	0.0466	0.0045	...	0.0089	0.0057	0.1428
18	3.896	2.693	0.444	0.1398	...	0.0495	0.0045	1.347	0.0093	0.0040	0.1395
19	3.903	0.0265
20	0.0276
21	3.878	2.713	0.440	0.1373	0.0265	0.0463	0.0045	1.341	0.0096	0.0043	0.1365
22	3.870	2.763	0.456	0.1456	...	0.0556	...	1.360	...	0.0044	0.1383
23	3.851	2.658	0.446	0.1382	0.0245	0.0495	0.0044	1.356	0.0092	0.0045	0.1388
24	...	2.659	0.462	0.0496	...	1.356	0.0103	0.0042	0.1493
25	...	2.714	0.447	0.1396	0.0264	0.0496	0.0043	1.345	0.0085	0.0039	0.1370
26	3.852	0.0269
27	0.1407	...	0.0484	0.0030	0.0066	...
28	0.450	...	0.0280	1.363
29	...	2.658
M_M	3.875	2.702	0.456	0.1438	0.0276	0.0501	0.0037	1.349	0.0087	0.0049	0.1397
s_M	0.021	0.038	0.012	0.0057	0.0016	0.0027	0.0007	0.017	0.0012	0.0012	0.0040
s_w	0.011	0.017	0.005	0.0014	0.0015	0.0009	0.0001	0.009	0.0004	0.0002	0.0027

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

Additional Information: Analysts No.2, 9 & 17 determined Co by ICP-MS (No 2) and ICP-OES (Nos. 9 & 17) reported 0.0026%, 0.0023% and <0.002% respectively. The same analysts used the same methods to determine Ti and found 0.020%, 0.018% and 0.017% and V, finding 0.009%, 0.008% and 0.014%.

CERTIFIED VALUES (C_v)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	As	Cu
C_v	3.875	2.702	0.456	0.1438	0.0276	0.0501	0.0037	1.349	0.0087	0.0049	0.1397
C(95%)	0.012	0.018	0.005	0.0027	0.0008	0.0012	0.0004	0.009	0.0006	0.0006	0.0019
Minimum Weight (g)	0.2	0.1	0.1	0.2	0.2	0.1	0.2	0.1	0.2	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 (C_v) by 10⁴ will yield the value in µg/g.

BCS-CRM No. 535

HIGH CARBON IRON POWDER

NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 3, 4, 7, 8, 13, 15 and 26 determined carbon using combustion/infra-red methods calibrated with carefully selected CRMs (see the traceability section for details) whilst Nos. 11, 18, 19, 21 and 22 used combustion/infrared methods but calibrated with pure chemicals: Analysts Nos. 16 and 17 used a gravimetric method after combustion in a stream of oxygen and No. 23 used a non-aqueous titration after combustion in a stream of oxygen.

SILICON

Analysts Nos. 3, 11, 16, 17, 18, 21, 22, 24 and 29 determined silicon gravimetrically, Nos. 18 and 22 dehydrating with hydrochloric acid (No. 18 followed ISO 439:1984) whilst the others dehydrated with perchloric acid; No.21 used IS 2308 (part VI:RA 2001), No. 24 used ASTM E350 and No. 29 digested in aqua regia. Analyst No. 8 used X-Ray Fluorescence Spectrometry (XRF), based on ISO 12677. The remaining analysts all used Inductively Coupled Optical Emission Spectrometry (ICP-OES); Nos. 4 and 5 after alkali fusion (No.4 dissolved the melt in HCl/HNO₃), Nos. 9 and 13 used microwave digestion (No. 13 digesting with aqua regia and HF), Nos. 12 and 14 digested with HCl/HNO₃/HF and No. 23 aqua regia and HF.

MANGANESE

With the exception of Analysts Nos. 2, 8, 16, 17 and 18 all Analysts determined manganese using ICP-OES. No. 2 used Inductively Coupled-Mass Spectrometry (ICP-MS), No. 8 XRF (based on ISO 12677), No. 16 Flame Atomic Absorption Spectrometry (FAAS) and Nos. 17 and 18 both used spectrophotometry, developing the colour with periodate, No. 17 following BS 6200:3.18.1:1985 and No. 18 ISO 629:1982. Of the Analysts using ICP-OES, No. 1, 9 and 14 used microwave digestion, No. 1 specifying an acid dissolution and No. 13 aqua regia with HF, No. 3 an acid digestion, No. 4 an alkali fusion and dissolution in HCl and HNO₃, No.5 used a combination of HCl, HNO₃, HF, HClO₄ and H₂O₂, No. 6 used ISO 11535:2006, No. 12, 21 and 23 used aqua regia No. 12 also using H₃PO₄/H₂SO₄; Analyst No. 14 used HCl, HNO₃ and HF, No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄, No. 22 followed ISO 10278:1996 and No. 28 BS EN 10355:2013.

PHOSPHORUS

Analysts Nos 2 and 27 determined phosphorus by ICP-MS, No. 27 using HCl, HNO₃ and HF. Analyst No. 8 used XRF, based on ISO 12677. Analysts Nos. 16, 17, 18 and 22 used the phosphovanadomolybdate photometric method. Nos 16, 17 and 18 after extraction with 4-methyl-pentan-2-one, No. 17 following BS EN 10184:1992, No. 18 ISO 2732:1984 and No. 22 ISO 10714:1992. The remaining analysts used ICP-OES, No. 3 after acid digestion, No. 4 after alkali fusion and dissolution in HCl and HNO₃, No. 5 used a combination of HCl, HNO₃, HF, HClO₄ and H₂O₂, No. 6 followed ISO 11535:2006, Nos. 9 and 13 used microwave digestion, Nos. 12, 13, 21 and 23 used aqua regia No. 13 also using HF. No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄

SULPHUR

Analysts Nos. 1, 3, 4, 7, 8, 13, 15 and 26 determined sulphur using combustion/infra-red methods calibrated with carefully selected CRMs (see the traceability section for details), Nos. 1 and 7 using a method based on ASTM E1019. Nos. 11, 19, 20 and 21 used combustion/infrared methods but calibrated with pure chemicals, Nos. 20 and 21 following ISO 4935. Analyst No.2 used ICP-MS, No. 16 burnt the sample in a stream of oxygen and titrated the resultant gases, No. 17 used a gravimetric method as BaSO₄ whilst Nos. 23, 25 and 28 all used ICP-OES.

CHROMIUM

Most analysts determined chromium by ICP-OES. No.3 used an acid digestion, No. 4 an acid dissolution in HCl and HNO₃, No. 5 a combination of HCl, HNO₃, HF, HClO₄ and H₂O₂, whilst No. 6 followed EPA 3052. Analysts Nos. 9 and 13 used microwave digestion (No. 13 digesting with aqua regia and HF), Nos. 12, 21 and 23 used aqua regia No. 12 also used H₃PO₄/H₂SO₄. Analyst No. 14 used HCl, HNO₃ and HF and No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄. No. 17 followed BS 6200:3.18.1:1985 and No. 24 ASTM E2594. Analysts Nos. 22 and 27 used ICP-MS, No. 27 with HCl, HNO₃ and HF. Analyst No. 12: used FAAS and No. 18 used the diphenylcarbazide photometric method in ISO 4936:1984.

MOLYBDENUM

With the exception of Analysts Nos. 2 and 27, who used ICP-MS (No. 27 after treatment with HCl, HNO₃ and HF), No. 11 who used FAAS and No. 18, who followed the photometric method with thiocyanate in ISO 4941, all the Analysts determined molybdenum with ICP-OES. Nos. 3 and 4 used an acid digestion, No. 4 specifying HCl and HNO₃, No. 6 followed EPA 3052 No. 9 used microwave digestion, Nos., 12, 21 and 23 used aqua regia No. 12 also using H₃PO₄/H₂SO₄. Analyst No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄, No. 17 followed BS EN 10355:2013.

NICKEL

Analyst No. 8 determined nickel by XRF, based on ISO 12677. Nos. 16, 18 and 28 used FAAS, No 18 in accordance with ISO 4940:1985. The remaining Analysts used ICP-OES. Nos.1, 9, 13 used microwave digestion, No.1 using unspecified acids and No 13 aqua regia and HF; No.4 an alkali fusion, dissolving the melt in HCl/HNO₃. Analyst No. 12, 21 and 23 used aqua regia No. 12 also using H₃PO₄/H₂SO₄; No. 14 used HCl, HNO₃ and HF and No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄. Analyst No. 24 followed ASTM E2594.

ALUMINIUM

Analyst No.2 used ICP-MS to determine aluminium whilst No 18 used FAAS, following ISO 9658. All the other Analysts used ICP.OES. No. 3 used an acid digestion whilst No. 4 used an alkali fusion and dissolved the melt in HCl/HNO₃. Analyst No. 5 used a combination of HCl, HNO₃, HF, HClO₄ and H₂O₂, No. 6 followed EPA 30527 and Nos. 12, 21 and 23 used aqua regia No. 12 also using H₃PO₄/H₂SO₄. No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄, No. 17 followed BS EN 10355:2013 and No. 24 ASTM E259

ARSENIC

Analysts Nos. 2 and 27 determined arsenic by ICP-MS, No. 27 using HCl, HNO₃ and HF. Nos. 11 and 18 used FAAS, No. 18 after ISO 1708:2004. The rest of the Analysts used ICP-OES; Nos. 3 and 4 used an acid digestion, No. 4 specifying HCl and HNO₃. No. 5 used a combination of HCl, HNO₃, HF, HClO₄ and H₂O₂, No. 6 followed EPA 30527 and No. 9 used microwave digestion whilst Nos. 12, 21 and 23 used aqua regia, No. 12 also using H₃PO₄/H₂SO₄. Analyst No. 15 dissolved with HCl and HNO₃ and fused any residue with Na₂CO₃, and No. 24 used ASTM E2823.

COPPER

Analysts Nos. 11, 16 and 18 determined copper using FAAS, No 18 following ISO 4943:1985. The other Analysts all used ICP-OES. No. 1, 9, 13 used microwave digestion, No. 1 an unspecified acid dissolution and No. 13 aqua regia and HF. Analyst No.3 used an acid digestion whilst No.4 used an alkali fusion and dissolved the melt in HCl/HNO₃). Analyst No.5 used a combination of HCl, HNO₃, HF, HClO₄ and H₂O₂, whilst Nos. 12, 21 and 23 used aqua regia, No. 12 also using H₃PO₄/H₂SO₄ 14 HCl, HNO₃ and HF. No. 15 dissolved with HCl and HNO₃ and fused any residue with KHSO₄, No. 17 followed BS EN 10355:2013 and No 24. ASTM E2594.

BCS-CRM No. 535

HIGH CARBON IRON POWDER

CO-OPERATING ANALYSTS

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2	RODUSHKIN, I.,	ALS Environmental AB, Lulea, Sweden.
3	BLUNDELL, P.,	ALS Inspection UK Ltd., Prescot.
4,	HURDITCH, P.,	AMG Analytical Services, Rotherham.
5	DERUITER, G.,	Alfred H Knight Ltd., Prescot.
6	VARADY, T. & KOVACS-SERES, B.,	Dunaferri Labor Nonprofit Ltd., Dunaujváros, Hungary.
7	COFFEY, R. N.,	Element Teesside Materials Testing, Middlesbrough.
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10	JIANG, J.,	Bureau Veritas, Shanghai, China.
11	CHATTOPADHYAY, S.,	Inspectorate Griffith India, Bhubaneswar, India.
12 & 29	KUBICZEK, M.,	Instytut Metalurgii Żelaza (IMZ), Gliwice, Poland.
13	FURNESS, W. & TUNNICLIFFE, M.,	Liberty Speciality Steels, Rotherham.
14 & 27	GRANFORS, M.,	Oy Narema AB., Närpiö, Finland.
15	BARTON, J., & CLARK, J.,	NSG European Technical Centre, Lathom.
16	ATKINSON, M. J.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
17 & 28	JONES, S. J.,	Ridsdale & Co Ltd., Middlesbrough.
18	POPOV, S. I.,	RZ Technical Control, Skopje, Republic of North Macedonia.
19	LINDKVIST, L.,	Kanthal AB, Hallstahammar, Sweden.
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24	CROOKS, M. & INGALSBE, J.,	Laboratory Testing Inc., Hatfield, PA, United States of America.
25	BULKA, V.,	Lithea s.r.o., Brno, Czech Republic.
26	TOMISSER, M.,	voestalpine Böhler Edelstahl GmbH & Co KG, Kapfenberg, Austria.

DESCRIPTION OF SAMPLE

BCS-CRM 535 is sold in the form of finely divided material passing a nominal 250 micron aperture and packed into bottles of 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 535 as defined in ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guide 35.

INTENDED USE

This sample 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 535 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. With the exception of C and S all laboratories used either stoichiometric analytical techniques or methods which were calibrated against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

For C and S, results have been obtained using stoichiometric analytical techniques, methods which were calibrated against pure stoichiometric compounds or by calibration using 5 certified reference materials carefully selected to be similar in composition to BCS-CRM 535 and all of which demonstrate full unbroken traceability to the SI. Different Analysts used different combinations of CRMs. The CRMs used were as follows: BCS-CRM 527 and ECRMs 478-2, 480-1, 481-1, 482-2, 484-1, 486-1, 487-2, 488-2, 489-1 and 490-1.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 535 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 on the bulk material using one way ANOVA and has been found to be acceptable, but, in any case, the material was mixed in a single batch, thus eliminating any inhomogeneity which may have existed at an earlier stage. It 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.

COMMUTABILITY

BCS-CRM 535 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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