



BUREAU OF ANALYSED SAMPLES LTD

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4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS BCS-CRM No. 377/6 IRON ORE SINTER

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN,
AUSTRALIA, CANADA, CHINA, HUNGARY, INDIA, THE NETHERLANDS AND SWEDEN
issued by the Bureau of Analysed Samples Ltd.

ANALYSES

Mean of 4 values - mass content in %.

Analyst	Fe	Si	Ca	Mg	Al	Ti	Mn	P	V	Cr	Zn	Pb	S	Na	K	F	Ni	Ba	Co	Cu
1	54.740	3.0320	5.8450	0.8973	0.7793	0.1040	0.5978	0.0558	0.0168	0.0163	1.0150	0.1500	0.5813	...	0.0085	0.0133	...	0.0825
2	54.809	2.9455	5.7285	0.9110	0.7493	0.1040	...	0.0578	0.0175	0.0163	0.9955	0.1451	...	0.1030	0.6073	0.2815
3	54.763	2.8946	5.6957	0.9358	0.8283	0.0930	0.5995	0.0593	0.0177	0.0177	1.0137	0.1508	0.0385	0.0994	0.5133	0.3208	0.0080	...	0.0016	0.0848
4	54.643	2.9636	5.8200	0.9320	...	0.1046	0.6035	...	0.0184	...	0.9846	0.1458	...	0.0981	0.5100
5	54.625	2.9866	5.7255	0.9354	0.7633	0.0948	0.6142	...	0.0175	0.0145	1.0210	0.1472	0.0404	0.0098	0.0943
6	...	2.9610	5.7818	0.8960	0.7703	0.0985	0.6017	0.0572	0.0179	0.0155	0.9908	0.1461	0.6307	...	0.0087	0.0093	0.0011	0.0844
7	54.888	2.9500	5.7625	0.8930	0.7508	0.1018	0.6085	0.0605	0.0168	0.0149	1.0215	0.1525	0.0448	...	0.6025	...	0.0084	0.0120	...	0.0738
8	54.800	2.9675	5.6425	0.8800	0.8150	0.0987	0.6275	...	0.0180	0.1468	...	0.0995	0.5123	0.2750
9	54.890	...	5.7850	0.8925	0.7725	0.1000	0.6125	0.0573	0.0180	0.0153	1.0075	0.1540	...	0.1095	0.6375	0.2975
10	54.731	3.0012	5.7233	0.8772	0.7669	0.0971	0.5907	0.0560	0.0184	0.0151	0.9928	0.1404	0.3042
11	54.648	2.9806	5.6597	0.8937	0.8030	0.1059	0.6046	0.0595	0.0197	0.0165	...	0.1467	...	0.0905	0.5039	0.3060
12	55.050	3.0383	5.8805	0.9458	...	0.0961	0.1100	0.5198
13	54.956	3.0391	5.7618	0.9192	0.8191	0.1050	0.6082	0.0602	0.0172	0.0146	0.9698	0.1439	...	0.1039	0.6250
14	0.8810	0.7744	0.0944	0.6013	0.0564	0.0176	0.0137	0.9745	0.1502	...	0.0958	0.5567
15	54.631	...	5.6680	0.9255	0.7517	...	0.5977	0.0592	0.0196	0.0136	1.0140	0.1566	...	0.0969	0.6185
16	54.578	3.0240	5.6695	...	0.8075	0.0988	0.5880	0.0608	0.0157	0.0152
17	54.973	2.9200	5.6608	0.8840	0.7833	0.0955	...	0.0610	...	0.0170	...	0.1518	...	0.0978	0.5575
18	...	3.0307	5.8424	0.9242	0.7900	0.1099	...	0.0596	0.0177
19	1.0068
20	1.0144
M_M	54.782	2.9823	5.7443	0.9073	0.7828	0.1001	0.6040	0.0586	0.0178	0.0154	1.0016	0.1485								
s_M	0.145	0.0450	0.0738	0.0227	0.0253	0.0049	0.0101	0.0019	0.0010	0.0012	0.0170	0.0043								
s_w	0.077	0.0108	0.0401	0.0063	0.0106	0.0025	0.0099	0.0013	0.0004	0.0010	0.0059	0.0023								

Values given in italics are for information only

Additional Information: Analyst No.1 determined Zr by ICP and reported a value of 0.0398%, Analyst No 3 reported a loss on ignition of 0.42% and a chloride level of <0.05%, Analyst No 5 determined C using combustion infra-red and found 0.472% and also reported 5.03% Fe(II) and Analyst No. 6 reported a value for Sn of 0.0064% and for As of 0.0067%, both obtained by XRF.

M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means. s_w: interlaboratory standard deviation.

CERTIFIED VALUES (C_v)

mass content in %

	Fe	Si	Ca	Mg	Al	Ti	Mn	P	V	Cr	Zn	Pb
C_v	54.78	2.982	5.74	0.907	0.783	0.1001	0.604	0.0586	0.0178	0.0154	1.002	0.1485
C(95%)	0.09	0.025	0.04	0.012	0.014	0.0025	0.006	0.0011	0.0006	0.0007	0.010	0.0024

The half width confidence interval $C(95\%) = \frac{t \times s_M}{\sqrt{n}}$ 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 35:2006 sections 6.1 and 10.5.2

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. 377/6

IRON ORE SINTER

NOTES ON METHODS USED

IRON

Analysts Nos. 1, 2, 7, 8 and 16 determined iron by X-Ray Fluorescence Spectrometry (XRF) using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 7 and 16 according to ISO 9516-1:2003. The remaining Analysts determined iron by titration with Cr (VI), all but Nos. 4 and 11 after reduction with Sn (II) No. 10 according to ISO 25971 and No 17 to IS 1493; Nos. 4 and 11 used Ti (III) to carry out the reduction, No. 4 following ASTM E1028 and No. 11 ISO 9507:1993.

SILICON

Analysts Nos. 1, 2, 6, 7, 8 and 16 determined silicon by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1: 2003. Analyst Nos. 3, 4, 11, 12, 13 and 17 used gravimetric methods involving the volatilisation of silicon with hydrofluoric acid; Nos. 3, 4 and 13 dehydrating with sulphuric acid, Nos.11 and 17 with hydrochloric acid, No. 11 according to ISO 2598:1993 and No.17 following IS 1493, and No. 12 with perchloric acid. Nos. 5 and 18 used Inductively Coupled Optical Emission Spectrometry (ICP-OES), No. 18 fusing the sample with a 2:1 mixture of sodium carbonate and borax. No. 10 used a photometric method, extracting silicon as molybdenum blue.

CALCIUM

Analysts Nos. 1, 2, 6, 7, 8 and 16 determined calcium by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1:2003. Analyst No. 3 used a gravimetric method, precipitating calcium as the oxalate. No. 4 separated calcium as the oxalate and titrated with permanganate. Analyst Nos. 5, 9, 10, 13, 15 and 18 used ICP-OES, No. 9 after fusion, No. 13 following ASTM E508-09 and No. 18 after fusing the sample with a 2:1 mixture of sodium carbonate and borax. Analysts Nos. 11, 12 and 17 titrated with EDTA, No. 17 according to IS 1493. Analyst No. 14 used Inductively-Coupled Plasma Mass Spectroscopy (ICP-MS) after microwave dissolution.

MAGNESIUM

Analysts Nos. 1, 2, 6, 7 and 8 determined magnesium by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6 and 7 according to ISO 9516-1:2003. Analysts Nos. 3, 5, 9, 10, 11, 13, 15, 17 and 18 used ICP-OES, No. 9 after fusion, No. 13 following ASTM E508-09 and No. 18 after fusing the sample with a 2:1 mixture of sodium carbonate and borax. Analyst No. 4 used Flame Atomic Absorption Spectrometry (FAAS) whilst No. 12 titrated with EDTA and No. 14 used ICP-MS after microwave dissolution.

ALUMINIUM

Analysts Nos. 1, 2, 6, 7, 8 and 16 determined aluminium by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1:2003. Analysts Nos. 3, 5, 9, 10, 11, 13, 15, 17 and 18 used ICP-OES, No. 9 after fusion, No. 13 according to IS 1493 (Part 4) 1988 and No. 18 after fusing the sample with a 2:1 mixture of sodium carbonate and borax. Analyst No. 14 used ICP-MS after microwave dissolution.

TITANIUM

Analysts Nos. 1, 6, 7, 8 and 16 determined titanium by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1:2003. Analysts Nos. 2, 3, 4, 5, 9, 10, 11, 13, 17 and 18 used ICP-OES, No. 2 with standard addition, No. 9 after fusion, No. 13 according to IS 1493 (Part 3) 1987 and No. 18 after fusing the sample with a 2:1 mixture of sodium carbonate and borax. Analyst No. 12 determined titanium photometrically with hydrogen peroxide whilst No. 14 used ICP-MS after microwave dissolution.

MANGANESE

Analysts Nos. 1, 4, 5, 9, 10, 11, 13 and 15 determined manganese by ICP-OES, No. 1 with microwave digestion, No. 9 after fusion and No. 13 according to IS 1493 (Part 3) 1987. Analyst No. 3 used FAAS whilst Nos. 6, 7, 8 and 16 used XRF with a fused bead, all but No. 8 according to ISO 9516-1:2003 and No. 14 used ICP-MS after microwave dissolution.

PHOSPHORUS

Analyst Nos. 1, 2, 6, 7 and 16 determined phosphorus by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1:2003. Analyst No. 3 used a photometric method, extracting the phosphovanadomolybdate complex with 4-methyl-pentan-2-one. Analysts Nos. 9, 10, 11, 13, 15, 17 and 18 used ICP-OES, No. 9 after fusion, No. 13 according to IS 1493(Part 3) 1987 and No. 18 after fusing the sample with a 2:1 mixture of sodium carbonate and borax. Analyst No. 14 used ICP-MS after microwave dissolution.

VANADIUM

With the exception of Analyst Nos.6, 7, 14 and 16 all Analysts determined vanadium by ICP-OES, No. 1 with microwave digestion, No. 2 using standard addition, No. 9 after fusion, No. 13 by following IS 1493 (Part 3) 1987 and No. 18 after fusing the sample with a 2:1 mixture of sodium carbonate and borax. Analysts Nos. 6, 7 and 16 used XRF with a fused bead, all according to ISO 9516-1:2003. Analyst No. 14 used ICP-MS after microwave dissolution.

CHROMIUM

Analyst Nos. 1, 2, 6, 7 and 16 determined chromium by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1:2003. With the exception of Analyst No. 14 the remaining Analysts used ICP-OES, No. 9 after fusion and No. 13 by following IS 1493 (Part 3) 1987. Analyst No. 14 used ICP-MS after microwave dissolution.

ZINC

Analysts Nos. 1, 6 and 7 determined zinc by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6 and 7 according to ISO 9516-1:2003. Analysts Nos. 2, 5, 9, 10, 13, 15, 19 and 20 used ICP-OES, No. 2 with standard addition, Nos. 9, 19 and 20 after fusion and No. 13 by following ISO 8753:1987. Analysts Nos. 3 and 4 used FAAS. Analyst No. 14 used ICP-MS after microwave dissolution.

LEAD

Analysts Nos. 1, 6, 7 and 8 determined lead by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6 and 7 according to ISO 9516-1:2003. Analysts Nos. 3 and 4 used FAAS. Analysts Nos. 2, 5, 9, 10, 11, 13, 15 and 17 used ICP-OES, No. 9 after fusion and No. 13 by following ISO 13311:1997. Analyst No. 14 used ICP-MS after microwave dissolution.

SULPHUR

Analyst No. 3 determined sulphur by redox titration following combustion. Analyst No 5 used combustion followed by infrared absorption. No. 7 used XRF with a fused bead according to ISO 9516-1:2003

SODIUM

Analysts Nos. 2, 6, 7 and 8 determined sodium by XRF using a fused bead, No. 6 using an in-house method based on ISO 9516-1:2003 whilst No. 7 followed ISO 9516-1:2003. Analysts Nos.3, 4 and 11 used FAAS. Analysts Nos. 9, 13, 15 and 17 used ICP-OES; No. 9 after acid digestion and No. 13 following ISO 6831:1986. Analyst No. 12 used flame photometry whilst No. 14 used ICP-MS after microwave dissolution.

POTASSIUM

Analysts Nos. 1, 2, 6, 7, 8 and 16 determined potassium by XRF using a fused bead, No. 1 according to BS EN ISO 12677:2011 and Nos. 6, 7 and 16 according to ISO 9516-1:2003. Analysts Nos. 3, 4 and 11 used FAAS. Analysts Nos. 9, 13, 15 and 17 used ICP-OES, No. 9 after acid digestion and No. 13 following ISO 6831:1986. Analyst No. 12 used flame photometry and No. 14 used ICP-MS after microwave dissolution.

FLUORINE

Analysts Nos. 2, 9 and 11 determined fluorine using a fluoride selective electrode, Nos. 9 and 11 after fusion with NaOH. Analyst No. 3 titrated with Th (IV) after separation of interfering ions. Analyst No.8 used XRF whilst No 10 determined fluorine photometrically with alizarin after pyrohydrolysis.

NICKEL

Analysts Nos. 1, 3 and 5 determined nickel by ICP-OES, No. 1 with microwave digestion. Analysts Nos.6 and 7 used XRF with a fused bead according to ISO 9516-1:2003

BARIUM

Analyst No. 1 determined barium by ICP-OES following microwave digestion. Analysts Nos. 6 and 7 used XRF with a fused bead according to ISO 9516-1:2003

COBALT

Analyst No. 3 determined cobalt using FAAS whilst No. 6 used XRF with a fused bead according to ISO 9516-1:2003

COPPER

Analysts Nos. 1 and 5 determined copper by ICP-OES, No. 1 with microwave digestion. Analyst No. 3 used FAAS whilst Analyst Nos. 6 and 7 used XRF with a fused bead according ISO 9516-1:2003

BCS-CRM No. 377/6

IRON ORE SINTER

CO-OPERATING ANALYSTS

1	SHAW, D.,	Lucideon, Stoke-on-Trent.
2	HURDITCH, P.,	AMG Superalloys UK. Ltd., Rotherham.
3, 19	CROCKER, F.H.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
4, 20	JONES, S.J., <i>BSc, CChem, MRSC,</i>	Ridsdale & Co. Ltd., Middlesbrough.
5	WOODHEAD I & SNOWDEN Y,	Tata Steel Testing Solutions, Scunthorpe
6	BIRCH S,	CSIRO Minerals, Glen Osmond, Australia
7	BOUCHARD M.,	Claisse (Corporation Scientifique) Quebec, Canada
8	CHEN, S.,	Ceram-Huaxia, Foshan City, China.
9	OOSTERHOFF, A.,	Laboratory Services International, Rotterdam, The Netherlands.
10	DE WEERDT, J.,	Tata Steel, IJmuiden, The Netherlands.
11	KONDOROSI, G.,	ISD Dunaferr, Dunaujvaros, Hungary.
12	FLORENCE, S.,	Mineral & Metallurgical Labs, Bangalore, India.
13, 18	UDPA, K. N., <i>PhD,</i>	Tata Steel Ltd., Jamshedpur, India.
14	RODUSHKIN, I.,	ALS Environmental, Lulea, Sweden.
15	HENRICH, A, <i>PhD,</i>	Höganäs AB, Höganäs, Sweden
16	DIDIC, M.,	LKAB, Malmberget, Sweden
17	MOHANASUNDARAM, N.,	Microlab, Chennai, India.

DESCRIPTION OF SAMPLE

BCS-CRM 377/6 is sold in the form of finely divided material passing a nominal 100 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 377/6 as defined in ISO Guide 34:2009 section 3.1, is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2009 and 35:2006.

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 a minimum sub-sample size of 1.0g. Users of this material should be aware that the use of a smaller sub-sample size will invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use.

STABILITY

BCS-CRM 377/6 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 377/6 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. 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 377/6 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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