BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

UKAS REFERENCE MATERIALS 4004

CERTIFICATE OF ANALYSIS
BCS/SS-CRM No. 117
Nb/Ti Interstitial Free Steel

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, AUSTRIA, CHINA, FINLAND, HUNGARY, INDIA, THE NETHERLANDS, POLAND, REPUBLIC OF NORTH MACEDONIA, RUSSIA, SPAIN and SWEDEN 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	Co	Cu	N	Nb	Pb	Sn	Ti	V
1	0.0011	...	0.1138	0.0102	0.0064	0.0195	0.00210	0.0196	...	...	0.00653	0.0152	0.00169	0.0187	...	...	0.0175	...
2	...	0.00242	0.1088	0.0070	0.0052	0.0187	0.00213	0.0205	0.0266	0.00145	0.00690	0.0144	...	0.0198	0.000069	0.00183	0.0167	0.00098
3	0.0019	0.00233	0.1073	0.0075	0.0054	0.0189	0.00190	0.0181	0.0295	0.00163	0.00578	0.0130	...	0.0174	...	0.00183	0.0167	0.00090
4	0.0020	0.00245	0.1158	0.0100	0.0056	0.0202	0.00203	0.0205	0.0283	0.00168	0.00575	0.0140	0.00175	0.0204	...	0.00165	0.0196	0.00098
5	0.0011	...	0.1123	0.0089	...	0.0208	0.00230	0.0213	0.0280	0.00193	0.00693	0.0163	...	0.0201	0.000050	0.00153	0.0193	...
6	0.0013	...	...	...	0.0062	...	...	...	...	...	...	...	0.00153	...	...	...	...	...
7	...	...	0.1140	0.0098	0.0048	0.0188	...	0.0190	0.0290	0.00200	0.00680	0.0168	...	0.0175	...	0.00100	0.0198	0.00093
8	...	0.00310	0.1121	0.0101	0.0063	0.0198	0.00230	0.0206	0.0287	0.00165	0.00603	0.0142	0.00105	0.0180	...	0.00170	0.0173	0.00140
9	...	0.00280	0.1175	0.0089	...	0.0205	0.00206	0.0196	0.0274	0.00191	0.00622	0.0147	...	0.0183	...	0.00137	0.0175	0.00096
10	0.0019	...	0.1153	0.0097	0.0061	0.0196	0.00206	0.0201	0.0294	0.00199	0.00709	0.0155	0.00134	...	...	0.00180	0.0171	0.00100
11	...	0.00365	0.1100	0.0094	0.0073	0.0190	0.00200	0.0180	0.0290	0.00178	0.00600	0.0160	...	...	...	0.00190	0.0170	0.00128
12	...	...	0.1213	0.0091	...	0.0216	...	0.0194	0.0278	...	0.00588	0.0141	...	0.0198	...	0.00075	0.0165	...
13	...	...	...	0.0096	...	0.0196	0.00185	0.0205	0.0313	0.00178	0.00645	0.0142	...	0.0187	...	...	0.0199	...
14	0.0020	.	...	...	0.0062	...	...	...	...	...	...	...	0.00143	...	...	...	...	...
15	...	...	0.1132	...	...	0.0204	0.00207	0.0202	0.0265	0.00129	0.00663	0.0149	...	0.0190	0.000099	0.00171	0.0173	...
16	0.0009	0.00383	0.1172	0.0091	0.0068	0.0200	0.00210	0.0199	0.0263	0.00148	0.00670	0.0149	...	0.0180	0.000100	...	0.0170	0.00103
17	0.0021	0.00263	0.1160	0.0094	0.0060	0.0191	0.00198	0.0196	0.0299	0.00158	0.00608	0.0148	0.00170	0.0173	...	0.00125	0.0172	0.00100
18	0.0018	0.00262	0.1104	0.0097	0.0061	0.0182	0.00225	0.0202	0.0281	0.00153	0.00613	0.0157	0.00185	0.0182	...	0.00152	0.0183	0.00116
19	0.0011	0.00275	0.1085	0.0083	0.0049	0.0175	0.00183	0.0185	0.0296	0.00183	0.00775	0.0172	0.00109	0.0210	...	0.00113	0.0174	0.00118
20	0.0012	0.00255	0.1159	0.0090	0.0061	0.0195	0.00230	0.0194	0.0271	0.00190	0.00675	0.0152	0.00148	0.0181	0.000063	0.00189	0.0169	0.00099
21	0.0014	...	...	0.0097	0.0062	...	...	...	0.0258	0.00190	...	...	0.00153	...	0.000069	0.00167	...	...
22	0.0016	0.00238	0.1167	0.0109	0.0064	0.0203	0.00205	0.0197	0.0306	0.00223	0.00813	0.0182	0.00148	0.0185	...	0.00120	0.0189	0.00111
23	...	0.00294	0.1151	0.0091	...	0.0195	0.00203	0.0195	0.0268	0.00179	0.00661	0.0144	...	0.0175	0.000059	0.00154	0.0166	0.00105
24	...	...	0.1113	0.0087	...	0.0188	0.00225	0.0189	...	...	0.00633	0.0137	...	0.0176	...	...	0.0160	0.00083
25	...	...	0.1144	...	0.0071	...	...	...	...	...	...	...	...	...	...	...	...	...
26	...	...	0.1157	0.0099	...	0.0200	...	0.0200	0.0294	...	...	0.0157	...	...	...	...	...	...
27	...	...	0.1182	...	...	...	...	0.0201	...	...	...	0.0155	...	...	...	...	...	...
28	...	0.00363	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.0165	0.00099
29	...	...	0.1134	...	...	0.0195	0.00176	0.0182	0.0267	...	...	0.0120	...	...	...	...	0.0143	0.00091
30	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.000063	...	...	...
31	...	...	...	...	...	...	...	...	...	...	...	...	0.00120	...	0.000055	...	...	...
32	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.000063	...	...	...
33	...	...	...	...	...	...	...	...	...	...	...	...	0.00160	...	...	...	...	...
M_M	0.0015	0.00286	0.1139	0.0093	0.0061	0.0196	0.00207	0.0196	0.0283	0.00175	0.00655	0.0150	0.00148	0.0186	0.000069	0.00152	0.0174	0.00104
s_M	0.0005	0.00051	0.0034	0.0009	0.0007	0.0009	0.00016	0.0009	0.0015	0.00023	0.00061	0.0013	0.00025	0.0011	0.000017	0.00033	0.0013	0.00014
s_w	0.0002	0.00033	0.0004	0.0007	0.0002	0.0004	0.00001	0.0003	0.0005	0.00014	0.00023	0.0003	0.00011	0.0004	0.000006	0.00007	0.0002	0.00001

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 Nos 2 & 29 reported Zn at 0.004% and 0.005% respectively using ICP-MS and ICP-OES.

CERTIFIED VALUES (Cv)
mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	As	Co	Cu	N	Nb	Pb	Sn	Ti	V
Cv	0.0015	0.00286	0.1139	0.0093	0.0061	0.0196	0.00207	0.0196	0.0283	0.00175	0.0065	0.0150	0.00148	0.0186	0.000069	0.00152	0.0174	0.00104
C(95%)	0.0003	0.00030	0.0015	0.0004	0.0004	0.0004	0.00008	0.0004	0.0007	0.00012	0.00028	0.0006	0.00014	0.0006	0.000013	0.00017	0.0006	0.00008
Minimum weight (g)	0.2	0.25	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.25	0.1	0.1	0.1	0.1	0.15

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.

Date of this edition: February 2024

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

BCS/SS-CRM 117
Nb/Ti Interstitial Free Steel
NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 3, 4, 6, 14 and 16 determined carbon using combustion/infra-red methods calibrated with carefully selected CRMs (see the Traceability section below for details). Analysts Nos. 17 and 18 determined carbon by non-aqueous titration after combustion in a stream of oxygen in accordance with BS 6200: 3.8.2: 1991. The remaining analysts used combustion/infrared methods calibrated with pure chemicals, Nos. 21 and 22 following ISO 9556.

SILICON

Analyst No. 2 determined silicon using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). Analysts Nos. 17 and 18 used the photometric method in BS 6200 3.8.2, No. 19 used a gravimetric method and the remaining analysts used Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES). No. 3 used an acid digestion, No. 4 calibrated using standard addition, No.9 used microwave digestion and No. 11 a four-acid dissolution. No. 22 used a nitric acid/hydrochloric acid mixture (5:1). No. 23 fused the sample with sodium carbonate and sodium tetraborate whilst No. 28 dissolved the material in a mixture of sulphuric and nitric acids.

MANGANESE

Most of the Analysts determined manganese by ICP-OES. Nos. 1 and 3 used an acid digestion, No. 4 calibrated using standard addition whilst No.7 followed the Chinese standard GB/T 20125:2006. Analyst No. 9 used microwave digestion, No. 11 a four-acid dissolution and No. 16 aqua regia. Analyst No. 19 followed EN 10351:2011, No. 22 used a nitric acid/hydrochloric acid mixture (5:1), whilst No.23 used a sulphuric acid/nitric acid digestion and No. 24 microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analysts Nos. 2, 5 and 15 used ICP-MS whilst Nos. 10 and 18 used a photometric method in which Mn is oxidised with potassium periodate and Nos. 12, 17 and 26 used Flame Atomic Absorption Spectrometry (FAAS).

PHOSPHORUS

Most Analysts determined phosphorus by ICP-OES. Nos.1, and 3 used acid dissolution, whilst No 7 followed the Chinese standard GB/T 20125:2006. Analyst No. 9 used microwave digestion and No. 11 used a four-acid digestion; Analyst Nos. 13 and 16 used aqua-regia, No.22 dissolved with hydrochloric and nitric acids, No. 23 used perchloric and nitric acids and No. 24 followed EN ISO 11885, after microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analysts Nos. 2, 5 and 21 used ICP-MS, Nos. 10, 17, 18 and 19 used photometric methods with phosphovanadomolybdate, Nos. 17, 18 and 19 using solvent extraction. Analyst No. 12 used a volumetric method, titrating ammonium phosphate and No. 20 also used a photometric method, based on molybdenum blue, after extraction.

SULPHUR

Analysts Nos. 1, 3, 4, 6, 8, 14 and 16 determined sulphur using combustion/infra-red methods calibrated with carefully selected CRMs (see the Traceability section below for details). Analyst No. 2 used ICP-MS and Nos. 7 and 25 used ICP-OES. Analyst No. 18 used an acid/base titration after combustion in a stream of oxygen and No. 8 used a gravimetric method, having added a known amount of S to all solutions. The remaining analysts used combustion/infrared methods calibrated with pure chemicals, No. 11 following the Chinese standard GB/T 20123-2006 and No. 21 and 22 ISO 4935.

CHROMIUM

Most Analysts determined chromium using ICP-OES. Analysts Nos. 2, 5 and 15 used ICP-MS, whilst No. 10 used a photometric method with diphenylcarbazide after separation of iron. Analysts Nos. 12 and 17 used FAAS. Of the Analysts who used ICP-OES Nos. 1 and 3 used an acid digestion, No. 4 calibrated using standard addition, No. 7 followed GB/T 20125:2006 and No. 9 used microwave digestion. No.11 used a four-acid dissolution, No. 13 aqua regia followed by fuming with sulphuric and phosphoric acids, No. 16 aqua regia and No. 17 followed EN 10351:201. Analyst No.22 dissolved with hydrochloric and nitric acids, No. 23 used perchloric and nitric acids and No. 24 followed EN ISO 11885, after microwave digestion with hydrochloric, nitric and hydrofluoric acids. No. 29 digested the sample at room temperature using nitric acid.

MOLYBDENUM

Other than Analysts Nos. 2, 5, 15 and 17 all of the Analysts determined molybdenum by ICP-OES. Nos. 1 and 3 used an acid digestion, No. 4 used standard addition, No. 9 used microwave digestion and No. 11 a four-acid dissolution. Analyst No. 13 used aqua regia followed by fuming with sulphuric and phosphoric acids whilst No. 16 used aqua-regia and Analyst No.22 hydrochloric and nitric acids. Analyst No. 23 used perchloric and nitric acids, No. 24 followed EN ISO 11885 and No. 29 digested the sample at room temperature using nitric acid. Analysts Nos. 2, 5 and 15 all used ICP-MS, No.15 after microwave digestion with Hydrochloric, nitric and hydrofluoric acids. No. 19 used a photometric method, in which the colour was developed with thiocyanate.

NICKEL

The majority of Analysts determined molybdenum with ICP-OES. Nos. 1 and. 3 used an acid digestion, No. 4 calibrated using standard addition, No. 7 followed GB/T 20125:2006 and No. 9 used microwave digestion. No.11 used a four-acid dissolution, No. 13 aqua regia followed by fuming with sulphuric and phosphoric acids, No. 16 aqua regia and No. 17 followed EN 10351:201. Analyst No.22 dissolved with hydrochloric and nitric acids, No. 23 used perchloric and nitric acids and No. 24 followed EN ISO 11885, after microwave digestion with hydrochloric, nitric and hydrofluoric acids. No. 29 digested the sample at room temperature using nitric acid. Analysts Nos. 2, 5 and 15 used ICP-MS, No. 15 using microwave digestion with hydrochloric, nitric and hydrofluoric acids. No. 12 used a photometric method with dimethylglyoxime whilst Analysts Nos. 12, 17 and 26 used FAAS.

ALUMINIUM

Analysts Nos. 2, 5, 15 and 21 determined aluminium with ICP-MS, No. 15 using microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analysts Nos. 12 and 19 used FAAS, whilst Analyst No. 26 used a photometric method with aluminon. The remaining analysts used ICP-OES. No. 3 used an acid digestion, No. 4 calibrated using standard addition, No. 7 followed GB/T 20125:2006 and No. 9 used microwave digestion. No.11 used a four-acid dissolution, No. 13 aqua regia followed by fuming with sulphuric and phosphoric acids, No. 16 aqua regia and No. 17 followed EN 10351:201. Analyst No.22 dissolved with hydrochloric and nitric acids, No. 23 used perchloric and nitric acids and No. 29 digested the sample at room temperature using nitric acid.

ARSENIC

Analysts Nos. 2, 5, 15 and 21 determined arsenic by ICP-MS. Analyst Nos. 17 and 19 used spectrophotometric methods, No. 17 using diethyldithiocarbamate and No. 19 molybdenum blue. The remaining Analysts used ICP-OES, No. 3 after acid digestion, No. 4 by standard addition, No. 7 following GB/T 20125:2006 and No. 9 after microwave digestion. No.11 used a four-acid dissolution, No. 13 aqua regia followed by fuming with sulphuric and phosphoric acids and No. 16 aqua regia. Analyst No.22 dissolved with hydrochloric and nitric acids whilst No. 23 used perchloric and nitric acids.

COBALT

Analysts Nos, 2, 5 and 15 determined cobalt by ICP-MS, No. 15 following microwave dissolution with hydrochloric, nitric and hydrofluoric acids. Analyst No. 12 used FAAS whilst the remaining Analysts used ICP-OES. Analyst Nos. 1 and 3 used an acid dissolution, No. 4 calibrated using standard addition, No. 7 used GB/T 2015:2006 and No. 9 used microwave digestion. Analyst No. 11 used a four-acid dissolution, No. 13 and 16 aqua-regia, No. 22 nitric and hydrochloric acids, No. 23 perchloric and nitric acids and No. 24 microwave digestion with hydrochloric and nitric acids.

BCS/SS-CRM 117
Nb/Ti Interstitial Free Steel

COPPER

Most Analysts determined copper using ICP-OES. Analyst Nos. 1 and 3 used acid dissolution. No. 4 used standard addition, No. 7 followed GB/T/ 2015:2006, No 9 used microwave digestion and No. 11 dissolved with four-acids. No. 13 used aqua-regia followed by fuming with sulphuric and phosphoric acids whilst No. 16 used aqua regia alone. Analyst No. 22 used nitric and hydrochloric acids, No. 23 perchloric and nitric acids and No. 29 digested with 16% nitric acid at room temperature. Analysts Nos. 2, 5 and 15 used ICP-MS, No. 15 after microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analyst No. 10 used a photometric method with diethyldithiocarbamate after solvent extraction. Analysts Nos. 12, 17 and 26 all used FAAS.

NITROGEN

Analysts Nos. 1, 4, 6, 8 and 14 determined nitrogen with thermal conductivity methods calibrated with carefully selected CRMs (see the Traceability section below for details). Analysts Nos. 17, 18 and 33 determined nitrogen by acid base titration following a Kjeldahl digestion. The other analysts used thermal conductivity calibrated with pure chemicals, No. 21 following ISO 10720.

NIOBIUM

All but four of the Analysts determined niobium by ICP-OES. Of these Analysts Nos. 1 and 3 used an acid digestion, No. 4 used standard addition, No. 7 used GB/T 20125:2006 and No. 9 microwave digestion. No. 13 used aqua-regia followed by fuming with sulphuric and phosphoric acids and No. 16 aqua regia. Analyst No. 22 digested with nitric and hydrochloric acids and No. 23 perchloric and nitric acids. Analysts Nos. 3, 5 and 15 determined niobium by ICP-MS, No. 15 after microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analyst No. 31 used a photometric method with 4-(2-pyridylazo)-2-napthol, according to ISO 9441:1988.

LEAD

Analysts Nos. 2, 5, 15, 21, 30 and 31 determined lead by ICP-MS, No. 15 after microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analysts Nos. 16 and 23 used ICP-OES, No. 23 using perchloric and nitric acids. Analyst Nos. 20 and 32 used electrothermal atomic absorption spectrometry (ETAAS).

TIN

Analysts Nos. 2, 5 15 and 21 determined tin using ICP-MS, No. 15 after microwave digestion with hydrochloric, nitric and hydrofluoric acids. Analyst No. 19 used ETAAS, following ISO 16918-1:2009. The remaining Analysts used ICP-OES, No. 3 after acid digestion and No. 4 calibrating with standard addition. Analyst No. 7 used GB/T 20125:2006, No. 9 used microwave digestion and No. 11 used a four-acid digestion. Analyst No. 22 digested with nitric and hydrochloric acids whilst No. 23 used a sodium carbonate/sodium tetraborate fusion.

TITANIUM

Most Analysts determined titanium with ICP-OES. Of these Nos. 1 and 3 used an acid digestion, No. 4 used standard addition, and No. 7 used GB/T 20125:2:2006. No. 9 used microwave digestion, No. 11 a four-acid dissolution and No. 13 used aqua-regia followed by fuming with sulphuric and phosphoric acids. Analyst No 16 used aqua-regia, No. 19 ISO 10280:1991, No. 22 digested with nitric and hydrochloric acids and No. 23 perchloric and nitric acids. Analyst No. 28 fused with sodium carbonate and sodium tetraborate whilst Analyst No. 29 digested at room temperature in 16% nitric acid. Analysts Nos. 2, 5 and 15 used ICP-MS, No. 15 using microwave digestion with hydrochloric, nitric and hydrofluoric acids. No. 19 used a photometric method with diantipyrylmethane described in ISO 10280:1991.

VANADIUM

Analyst No.2 determined vanadium using ICP-MS whilst Analyst No. 19 used FAAS. The remaining Analysts all used ICP-OES, No. 4 calibrating using standard addition. Analyst No. 7 used GB/T 20125:2006, No. 8 microwave digestion. No. 11 a four-acid digestion and No. 16 aqua-regia. Analyst No. 22 digested with nitric and hydrochloric acids whilst No. 23 used a sodium carbonate/sodium tetraborate fusion. No. 28 used a perchloric acid/nitric acid mixture whilst No. 29 digested with 16% nitric acid at room temperature.

CO-OPERATING ANALYSTS

1	QUIRÓS, E. & PÉREZ, D.,	Acerinox Europa S.A.U., Cadiz, Spain.
2	RODUSHKIN, I.,	ALS Environmental AB, Lulea, Sweden.
3	BLUNDELL, P.,	ALS Inspection UK, Prescot.
4	HURDITCH, P.,	AMG Analytical Services, Rotherham.
5	SHAW, T.,	AWE, Aldermarston.
6	SNOWDON, Y.,	British Steel, Scunthorpe.
7	KONDOROSI, G.,	Dunaferr Labor Nonprofit Ltd., Dunaújváros, Hungary.
8	COFFEY, R. N.,	Element Teesside Materials Testing, Middlesbrough.
9	HENRICH, A.,	Höganäs, Sweden AB, Höganäs, Sweden.
10, 26 & 27	STEPANOVSKIKH, V.,	Institute of Certified Reference Materials (ICRM), Yekaterinburg, Russia.
11	JIANG, J.,	Bureau Veritas, Shanghai, China.
12	CHATTOPADHYAY, S.,	Inspectorate Griffith India, Bhubaneswar, India.
13	KUBICZEK, M.,	Instytut Metalurgii Żelaza (IMZ), Gliwice, Poland.
14	FURNESS, W. & TUNNICLIFFE, M.,	Liberty Speciality Steels, Rotherham.
15	GRANFORS, M.,	Oy NAREMA Ab, Närpiö, Finland.
16	BARTON, J. & CLARK, J.,	NSG European Technical Centre, Lathom.
17 & 33	ATKINSON, M. J.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
18 & 25	JONES, S. J.,	Ridsdale & Co Ltd., Middlesbrough.
19	POPOV, S.I.,	RZ Tehnicka Control AD, Skopje, Republic of North Macedonia.
20 & 32	LINDKVIST, L.,	Kanthal, Hallstahammar, Sweden.
21 & 30	CARLSTRÖM-WÄNGELIN, A.,	Alleima Tube AB, Sandviken, Sweden.
22	MOHAPATRA, J. K.,	Tata Steel Limited, Jamshedpur, India.
23 & 28	KONIJN, A.,	Tata Steel Europe, IJmuiden, The Netherlands.
24	GRIENBERGER, S.,	voestalpine Stahl GmbH, Linz, Austria.
29	BARNES, P. & DAWE, A,	Tata Steel Europe, Port Talbot.
31	MANDAVA, V.,	Lucid Laboratories PVT Ltd., Hyderabad, India.

BCS/SS-CRM 117
Nb/Ti Interstitial Free Steel

DESCRIPTION OF SAMPLE

British Chemical Standard BCS-CRM 117 is sold in the form of chips passing a nominal 1700µm aperture sieve from which the fines passing a nominal 250µm aperture sieve have been removed. It is supplied in bottles containing 100g.

Spectroscopic Standard SS-CRM 117 is sold in the form of 44mm diameter discs.

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/SS-CRM 117 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 117 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.

SS-CRM 117 is intended for establishing and checking the calibration of Optical Emission and X-Ray Spectrometers for the analysis of similar materials. The "as received" working surface of the sample should be linished before use to remove any protective coating. An area 6mm in diameter in the centre of the disc should be avoided for optical emission spectrometry. Users of optical emission spectrometry should also be aware that a minimum of three sparks, made on separate, clean locations across the face of the disc, should be obtained in order to establish a reliable mean value.

STABILITY

BCS-CRM 117 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.

SS-CRM 117 will remain stable provided that it is not subjected to excessive heat (e.g., during preparation of the working surface).

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, S and N 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, S and N 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/SS-CRM 117 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-CRMs 111, 111/1, 115, 116, 431/2 and 432/2 and ECRMs 055-2, 056-2, 057-2, 064-1, 085-1, 088-2, 090-1 and 097-2.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS/SS-CRM 117 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 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 117 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.

In the case of SS-CRM 117 it has been established that, when using optical emission spectrometers, materials of similar composition from different sources may respond differently. The user should be aware that the metallurgical history of this SS-CRM may not accurately reflect the metallurgical history of the user's own materials.

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
February 2024