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4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS/SS-CRM No. 479
NIOBIUM STABILISED STAINLESS STEEL

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, AUSTRIA, HUNGARY, INDIA, 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	Ni	Cu	N	Nb	Ti	V
1	…	0.5793	0.6760	0.00365	…	19.9350	24.9025	0.0059	…	…	0.0313	…
2	0.0533	…	…	…	0.00265	…	…	…	0.00579	…	…	…
3	…	0.5583	0.6873	0.00240	0.00368	…	…	0.0046	…	0.6035	0.0306	0.0046
4	0.0538	0.5303	0.6788	…	0.00290	…	24.7542	0.0054	0.00563	…	…	0.0051
5	…	…	0.6778	0.00320	…	…	…	0.0064	…	0.6440	0.0300	0.0056
6	…	0.5453	0.6778	0.00288	…	19.9326	24.9767	0.0052	…	0.6350	0.0281	0.0048
7	…	…	0.6793	0.00238	…	19.9658	24.8105	0.0053	…	0.6169	0.0292	0.0042
8	0.0539	0.5695	0.6888	0.00283	0.00300	19.9098	24.8720	…	0.00562	0.6367	0.0311	0.0055
9	0.0526	0.5561	0.6823	0.00373	0.00292	19.9350	24.9450	…	0.00553	0.6493	0.0300	…
10	0.0532	…	0.6874	…	0.00326	…	…	…	0.00575	0.6108	0.0311	…
11	…	0.5548	0.6789	0.00258	…	19.9203	…	0.0050	…	0.6222	0.0305	0.0055
12	0.0534	0.5550	0.6665	0.00238	0.00229	19.9636	24.7633	0.0043	0.00535	0.6285	0.0277	0.0052
13	…	0.5328	…	0.00300	…	19.9002	24.8978	0.0059	…	0.6018	0.0303	…
14	0.0510	0.5555	0.6765	0.00350	0.00310	19.9144	24.8258	0.0054	0.00545	0.6089	0.0333	0.0060
15	0.0499	0.5686	0.6676	0.00317	0.00356	19.9248	24.7902	0.0043	0.00533	0.6516	0.0311	0.0051
16	0.0515	0.5491	…	0.00258	0.00305	19.8564	24.7748	0.0050	0.00643	0.6247	0.0322	0.0056
17	0.0524	…	0.6985	…	0.00288	19.9439	24.9317	0.0047	0.00560	0.6006	0.0299	0.0064
18	0.0531	0.5645	0.6731	0.00270	0.00286	19.8724	25.1111	0.0058	0.00552	0.6530	0.0321	0.0040
19	0.0564	…	…	…	0.00255	…	…	…	0.00520	…	…	…
20	0.0534	…	…	…	0.00338	…	…	…	0.00583	…	…	…
21	0.0533	…	…	…	…	…	…	…	0.00586	…	…	…
22	…	…	…	…	…	…	…	…	…	…	…	0.0052
23	0.0529	…	…	…	0.00314	…	…	…	0.00568	…	…	…
24	…	…	…	…	…	…	…	…	…	…	…	0.0052
25	…	…	…	…	…	19.9408	24.8040	…	…	…	…	…
26	…	…	…	…	…	…	…	…	0.00554	…	…	…
27	0.0523	…	…	…	0.00291	…	…	…	0.00605	…	…	…
28	…	…	…	…	…	…	…	…	…	0.6129	0.0320	…
29	…	0.5193	…	…	…	19.9091	…	…	…	…	…	…
M_M	**0.0529**	**0.5527**	**0.6798**	**0.00293**	**0.00301**	**19.9216**	**24.8685**	**0.0052**	**0.00566**	**0.6250**	**0.0306**	**0.0052**
s_M	0.0015	0.0165	0.0083	0.00047	0.00036	0.0300	0.1004	0.0007	0.00030	0.0183	0.0015	0.0007
s_w	0.0004	0.0034	0.0039	0.00013	0.00015	0.0323	0.0656	0.0002	0.00015	0.0045	0.0005	0.0002

Mo	*Al*	*As*	*B*	*Co*	*Pb*	*Sn*	*W*	*Ta*
0.0031	…	…	*0.0002*	*0.0025*	*0.00013*	…	…	*0.0038*
…	…	…	…	…	…	…	…	…
0.0023	*0.0120*	*0.0014*	*<0.0001*	*0.0012*	*0.00005*	*0.0004*	*0.0001*	*0.0007*
0.0025	*0.0116*	…	*<0.0001*	*0.0032*	…	*<0.0001*	*0.0008*	*<0.001*
0.0022	*0.0103*	*0.0009*	*0.0002*	*0.0013*	*<0.000002*	*0.0003*	*0.0001*	*0.0030*
0.0021	*0.0122*	*0.0012*	…	*0.0013*	*<0.0001*	*<0.001*	*<0.001*	*0.0007*
0.0021	*0.0111*	*0.0011*	*<0.001*	…	*0.00003*	*0.0005*	…	…
0.0030	*0.0154*	*0.0017*	*0.0004*	*0.0024*	…	*<0.001*	*0.0043*	*0.0051*
0.0051	*0.0147*	…	*<0.0005*	*<0.0015*	…	*<0.001*	*0.0018*	*<0.001*
0.0046	…	…	…	…	…	…	…	…
0.0025	*0.0129*	*0.0019*	*0.0003*	*0.0021*	*0.00060*	*0.0009*	*0.0026*	*0.0026*
0.0029	*0.0146*	*0.0023*	*0.0003*	*0.0004*	…	*0.0006*	*0.0010*	*<0.0002*
0.0055	*0.0103*	*0.0021*	…	*0.0019*	*0.00040*	*0.0018*	…	…
0.0012	*0.0166*	*0.0015*	*<0.0005*	*0.0015*	*<0.0005*	*0.0016*	…	*0.0034*
<0.0001	*0.0102*	*0.0010*	*0.0001*	*0.0014*	*<0.001*	*<0.0004*	…	…
0.0022	*0.0137*	*0.0008*	*0.0005*	*0.0017*	*<0.0001*	*0.0004*	…	…
…	*0.0197*	…	…	…	…	…	…	…
0.0025	*0.0164*	…	…	…	*0.00009*	…	*0.0019*	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	*0.0007*
…	…	…	…	…	…	…	…	…
…	*0.0123*	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…
0.003	*0.013*	*0.002*	*<0.0005*	*0.002*				

Values given in italics are for information only

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

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Ni	Cu	N	Nb	Ti	V
Cv	**0.0529**	**0.553**	**0.680**	**0.0029**	**0.0030**	**19.922**	**24.87**	**0.0052**	**0.0057**	**0.625**	**0.0306**	**0.0052**
C(95%)	0.0008	0.010	0.005	0.0003	0.0002	0.017	0.06	0.0004	0.0002	0.010	0.0008	0.0004

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

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NIOBIUM STABILISED STAINLESS STEEL

NOTES ON METHODS USED

CARBON

Analysts Nos. 2, 4, 8, 9, 10, 12, 18 and 20 determined carbon by combustion/infra-red methods calibrated with carefully selected CRMs (see below for details). Analysts Nos. 14 and 15 used a non-aqueous titration following combustion, and Analysts Nos. 16, 17, 19, 21, 23 and 27 used combustion infrared methods calibrated with pure chemicals or stoichiometric compounds.

SILICON

Analysts Nos. 1, 4, 9, 11, 12, 13, 16 and 18 determined silicon by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). Analyst No. 3 used Inductively Coupled Plasma Mass Spectrometry (ICP-MS) whilst Analysts Nos. 6, 8 14 and 15 used a gravimetric method, dehydrating with perchloric acid. Analyst No. 29 used the molybdenum blue photometric method.

MANGANESE

All Analysts except for Nos. 3, 7 and 15 determined manganese by ICP-OES. Analyst No. 3 used ICP-MS, No. 7 Flame Atomic Absorption Spectrometry (FAAS) whilst No.15 determined manganese photometrically after oxidation with periodate.

PHOSPHORUS

Analysts Nos. 1, 5, 7, 8, 9, 11, 12, 13 and 18 determined phosphorus by ICP-OES. Analyst No. 3 used ICP-MS; Analysts Nos. 6, 14, 15 and 16 used a photometric method, extracting the phosphovanadomolybdate complex with 4-methyl-pentan-2-one.

SULPHUR

Analysts Nos. 2, 4, 8, 9, 10, 12, 18 and 20 determined sulphur by combustion/infra-red methods calibrated with carefully selected CRMs (see below for details). Analyst No. 3 used ICP-MS, No. 14 used an acidimetric titration after combustion, No. 15 used ICP-OES and Analysts Nos. 16, 17, 19, 23 and 27 used combustion infrared methods calibrated with pure chemicals or stoichiometric compounds.

CHROMIUM

Analysts Nos. 1, 9, 11, 12, 13, 18, 25 and 29 determined chromium by ICP-OES. With the exception of Lab 16 the remaining Analysts determined chromium volumetrically by titration with Fe (II) after oxidation with persulphate. Analyst No. 16 used the indirect titration method of ISO 15355:1999.

NICKEL

Analysts Nos. 1, 4, 6, 9, 12, 18 and 25 determined nickel by ICP-OES. Analysts Nos. 7, 13, 16, and 17 used a gravimetric method, precipitating nickel with dimethylglyoxime, No. 8 used a cyanometric titration, Nos. 14 and 15 also used dimethylglyoxime to precipitate nickel, No. 14 then titrating with EDTA and No.15 with dichromate.

COPPER

All Analysts, except for Nos. 3, 7, 12, 14 and 15 determined copper with ICP-OES. Analysts Nos. 3 and 12 used ICP-MS and Nos. 7, 14 and 15 used FAAS.

NITROGEN

Analysts Nos. 2, 4, 8, 9, 10, 12, 18 and 20 determined nitrogen by thermal conductivity after decomposition in a graphite crucible and calibrated with carefully selected CRMs (see below for details). Analyst No. 14 used a photometric method with Nessler's reagent following distillation. Nos. 15 and 16 used an acid base titration following distillation and Analysts Nos. 17, 19, 21, 23, 26 and 27 used thermal conductivity after decomposition in a graphite crucible and calibrated with pure chemicals or stoichiometric compounds.

NIOBIUM

All Analysts, except for No 3, determined niobium by ICP-OES. Analyst No. 3 used ICP-MS.

TITANIUM

All Analysts, except for Nos. 3 and 15, determined titanium by ICP-OES. Analyst No. 3 used ICP-MS and No. 15 used a photometric method with diantipyrylmethane.

VANADIUM

All Analysts, except for Nos. 3, 5, 12 and 24 determined vanadium by ICP-OES. Analysts Nos. 3, 5 and 12 used ICP-MS whilst No. 24 used FAAS.

MOLYBDENUM

All Analysts, except for Nos. 3 and 7, determined molybdenum by ICP-OES. Analyst No. 3 used ICP-MS and No. 7 FAAS.

ALUMINIUM

All Analysts, except for Nos. 3 and 24, determined aluminium by ICP-OES. Analyst No. 3 used ICP-MS and No. 24 FAAS.

ARSENIC

Analysts Nos. 3, 12 and 16 determined arsenic by ICP-MS. Analysts Nos. 5 and 7 used GFAAS, No. 6 used FAAS with hydride generation, No. 8 used ICP-OES with hydride generation, Nos. 11 and 13 used ICP-OES whilst Nos. 14 and 15 used a photometric method with silver diethyldithiocarbamate.

BORON

All Analysts, except for Nos. 3, 8, 15 and 16, used ICP-OES. Analyst No. 3 used ICP-MS and Nos. 8, 15 and 16 used a photometric method with curcumin.

COBALT

All Analysts except for Nos. 3, 5 and 15 determined cobalt using ICP-OES. Analyst No. 3 used ICP-MS whilst Nos. 5 and 15 used FAAS.

LEAD

Analysts Nos. 1, 11, 13 and 14 used ICP-OES. Analysts Nos. 3, 5 and 16 used ICP-MS; No. 6 used FAAS extracting with tri-octylphosphine oxide, potassium iodide and methylisobutylketone; No. 7 used GFAAS and Nos. 15 and 18 used FAAS.

TIN

All Analysts, except for Nos. 3, 5, 6, 7 and 16, determined tin by ICP-OES. Analyst Nos. 3, 5 and 16 used by ICP-MS whilst No. 6 used FAAS with hydride generation and No. 7 used GFAAS.

TUNGSTEN

All Analysts, except for Nos. 3 and 12 used ICP-OES. Analysts Nos. 3 and 12 used ICP-MS.

TANTALUM

All Analysts, except for Nos. 3, 12 and 22 determined tantalum by ICP-OES. The remaining Analysts used ICP-MS.

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CO-OPERATING ANALYSTS

1	PINOS, E. Q.,	Acerinox, Cadiz, Spain.
2	FARLEY, E. J & CHEETHAM, D.,	Alcoa Howmet, Exeter.
3	RODUSHKIN, I.,	ALS Environmental, Lulea, Sweden.
4	BLACKWELL, M., & WEBSTER, M.,	ATI Specialty Materials, Sheffield.
5, 22	COLE, P. & WILSON C.,	ATI Specialty Materials, Monroe, USA.
6	PRATTES, K.,	Böhler Edelstahl GmbH & Co KG, Kapfenberg, Austria.
7, 24	POLINKO, C.,	Carpenter Technologies, Reading, USA.
8	HASTINGS, J.,	Element Materials Testing, Sheffield.
9	COFFEY, R, N., *BSc, (Hons)*	Exova Teesside, Middlesbrough.
10, 23, 26	GUSTAVSSON, I	Gustavsson Consulting, Stockholm, Sweden.
11	HENRICH, A, *PhD,*	Höganäs AB, Höganäs, Sweden.
12, 19	SCRIMSHIRE, P. J.,	IncoTest, Hereford.
13	KONDOROSI G.,	ISD Dunaferr, Dunaújváros, Hungary.
14	CROCKER, F. H.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
15, 29	JONES, S.J., *BSc, CChem, MRSC*	Ridsdale & Co Ltd, Middlesbrough.
16	WICHARDT, C.,	AB Sandvik Materials Technology, Sandviken, Sweden.
17, 25	UDPA, K.N.,	Tata Steel, Jamshedpur, India.
18	RUGG, P.,	Tata Steel, Stocksbridge.
20, 21	LIND, A.,	Chemal, Hofors, Sweden.
27	LINDKVIST, L.,	Sandvik Heating Technology AB, Hallstahammar, Sweden.
28	PETTERSSON, J.,	Uppsala University, Uppsala, Sweden.

DESCRIPTION OF SAMPLE

BCS-CRM 479 is sold in the form of chips passing a nominal 1700µm aperture sieve from which the fines passing a nominal 250µm sieve have been removed. It is supplied in bottles containing 100g,

SS-CRM 479 is sold in the form of 38mm 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-CRM 479 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

BCS-CRM 479 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.

SS-CRM 479 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. It 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.

With the exception of C, S and N 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.

For C, S and N at least half of the results have been obtained using either stoichiometric analytical techniques or methods which were calibrated against pure metals or stoichiometric compounds. The remaining results have been obtained using calibrations based on 5 certified reference materials carefully selected to be similar in composition to BCS-CRM 479 and all of which demonstrate full unbroken traceability to the SI. Different Analysts used different combinations of CRMs. The CRMs used were as follows:

C: BCS-CRM 332, BCS-CRM 465/1, BCS-CRM 467/1, BCS-CRM 474, BCS-CRM 475, ECRM 231-2, ECRM 237-1, ECRM 281-1, ECRM 285-2, ECRM 292-1.

S: BCS-CRM 461/1, BCS-CRM 462/1, BCS-CRM 465/1, BCS-CRM 466/2, ECRM 226-1 ECRM 285-2, ECRM 292-1, ECRM 297-1, JK 37.

N: BCS-CRM 465/1, BCS-CRM 466/2, ECRM 231-2, ECRM 281-1, ECRM 284-2, ECRM 285-2, ECRM 288-1, ECRM 297-1.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS/SS-CRM 479 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 of the finely divided material (BCS-CRM 479) has been assessed on the bulk material using one way ANOVA and has been found to be acceptable. Homogeneity of the discs (SS-CRM 479) 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 479 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 479 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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