MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD.

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS*/SS†-CRM No. 350
NICKEL ALLOY IN 713

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

The material from which this CRM was prepared was cast by Howmet Alloys International, Exeter and subjected to Hot Isostatic Pressing

CO-OPERATING ANALYSTS AND FIRMS

INDEPENDENT ANALYST

1. PAGE-GIBSON, J.E. *BSc, CChem, MRSC,* Ridsdale & Co Ltd., Middlesbrough.

GOVERNMENT DEPARTMENT

2. FRYER, R.E.J., *BSc, PhD, CChem, MRSC,* Directorate of Quality Assurance/Technical Support, Ministry of Defence, London.

ANALYSTS representing MANUFACTURERS and USERS

3. COX, R.J, Rolls Royce plc, Derby.
4. DEARNALEY, P., Firth Rixson plc., (Glossop Superalloys), Glossop
5. HAIRE, R.J., Howmet Corporation, Alloy Division, New Jersey, USA.
6. HANCOCK R., *BMet, CEng, MIM* Stocksbridge Engineering Steels, Sheffield.
7. KUNISH,R., & MARCZAK, G.S., Howmet Corporation, Corporate Quality Assurance, Michigan, USA.
8. LEE, J.D., *BA , MSc, LRSC, MIQA* & BATTY, G., Ross & Catherall Ltd., Killamarsh.
9. SHAW, D.H., Howmet Alloys International, Exeter
10. THORNTON, K., *BA* Inco Alloys Ltd., Hereford

ANALYSES

Mean of 4 values – mass content in %.

Analyst No.	C	Si	Mn	Cr	Mo	Ni	Al	B	Co	Nb	Ti	W	Zr	Fe
1	0.134	0.104	0.021	13.37	4.22	70.5	6.04	0.011	0.345	2.12	0.89	0.091	0.080	1.50
2	0.138	...	0.016	13.38	4.30	71.0	6.08	0.013	0.334	2.17	0.88	0.093	0.070	1.50
3	0.142	0.100	...	13.44	4.22	70.4	5.90	0.011	0.333	2.22	0.88	0.089	0.072	1.49
4	0.138	...	0.018	13.46	4.34	70.8	...	0.012	0.330	...	...	...	...	1.47
5	0.135	0.116	0.019	13.47	4.29	...	5.93	0.013	0.340	2.18	0.88	0.102	0.071	1.48
6	0.138	0.118	0.018	13.33	4.24	70.6	5.91	0.012	0.340	2.12	0.85	0.103	0.080	1.53
7	0.142	0.128	0.019	13.37	4.27	...	5.97	0.014	0.346	2.18	0.86	0.090	0.060	1.47
8	0.138	...	0.019	13.50	4.36	70.9	...	0.014	0.337	2.21	...	...	...	1.55
9	0.136	0.108	0.020	13.50	4.40	...	5.96	0.014	...	2.16	0.85	...	...	1.54
10	0.135	0.097	0.019	13.44	4.28	71.1	5.95	0.012	0.334	2.17	0.88	0.092	0.069	1.51
M_M	**0.138**	**0.110**	**0.019**	**13.43**	**4.29**	**70.8**	**5.97**	**0.013**	**0.338**	**2.17**	**0.87**	**0.094**	**0.072**	**1.50**
s_M	0.003	0.011	0.002	0.06	0.06	0.3	0.07	0.002	0.006	0.04	0.02	0.006	0.007	0.03
s_W	0.001	0.005	0.001	0.03	0.03	0.2	0.03	0.001	0.005	0.05	0.01	0.004	0.002	0.02

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

The above figures are those which each Analyst has decided upon after careful verification

Additional Information: Ta approximately 0.05%

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	Cr	Mo	Ni	Al	B	Co	Nb	Ti	W	Zr	Fe
Cv	**0.138**	**0.110**	**0.019**	**13.43**	**4.29**	**70.8**	**5.97**	**0.013**	**0.338**	**2.17**	**0.87**	**0.094**	**0.072**	**1.50**
C(95%)	0.002	0.011	0.001	0.05	0.05	0.3	0.06	0.001	0.005	0.03	0.02	0.006	0.007	0.02

The half width confidence interval **C**(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate Student's t value and "n" is the number of acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:1989 section 4.

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NOTES ON METHODS USED

CARBON

Analyst No. 1 determined carbon by combustion and non-aqueous titration. All other Analysts used high frequency combustion and infrared absorption.

SILICON

Analysts Nos. 1 and 6 determined silicon gravimetrically after dehydration, No. 1 with sulphuric acid and No. 6 with perchloric acid. Nos. 3, 5, 7 and 10 determined silicon using Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) and No. 9 used Flame Atomic Absorption Spectrometry (FAAS).

MANGANESE

Analyst No. 1 determined manganese photometrically after oxidation with periodate. Nos. 2, 5, 7 and 10 used ICP-AES. Nos. 4, 6, 8 and 9 used FAAS.

CHROMIUM

All Analysts, except Nos. 5 and 10, determined chromium titrimetrically with ammonium ferrous sulphate after oxidation with persulphate/silver nitrate. Nos. 5 and 10 used ICP-AES.

Analyst No. 10 also used the titrimetric method and obtained a mean value of 13.36%.

MOLYBDENUM

Analysts Nos. 1, 2, 3, 6 and 8 determined molybdenum photometrically as the oxythiocyanate after extraction of the complex. Nos. 4 and 9 used FAAS and Nos. 5, 7 and 10 used ICP-AES.

Analyst No. 10 also used the photometric method without extraction and obtained a mean value of 4.28%.

NICKEL

Analysts Nos. 1, 2, 3, 4, 6 and 8 determined nickel after precipitation with dimethylglyoxime. No. 1 dissolved the precipitate in dilute sulphuric acid, boiled with excess of ferric sulphate and titrated the ferrous salt formed with dichromate (Analoid Method No. 62). Nos. 2, 3, 4 and 6 titrated with EDTA. No. 8 determined nickel gravimetrically. No. 10 used ICP-AES.

ALUMINIUM

Analysts Nos. 1, 6 and 9 determined aluminium by FAAS, No. 6 after mercury cathode separation. Nos. 2 and 3 also used mercury cathode separation, No. 2 titrated with EDTA and No. 3 determined aluminium gravimetrically with oxine after separating titanium with cupferron. Nos. 5, 7 and 10 used ICP-AES.

BORON

Analysts Nos. 1, 2, 3, 4, 6, 8, 9 and 10 determined boron photometrically, all used curcumin, except No. 8 who used dianthrimide. Nos. 5 and 7 used ICP-AES.

COBALT

Analysts Nos. 1 and 3 determined cobalt photometrically with nitroso-R-salt. Nos. 2, 4, 6 and 8 used FAAS. Nos. 5, 7 and 10 used ICP-AES.

Analyst No. 1 also determined cobalt using FAAS and obtained a mean value of 0.345%.

NIOBIUM

Analysts Nos. 1, 8 and 9 determined niobium photometrically with 4-(2-pyridylazo)-resorcinol (PAR). Nos. 2, 3, 5, 7 and 10 used ICP-AES. No. 6 determined niobium gravimetrically after magnesia separation and correcting for titanium.

Analyst No. 10 also determined niobium gravimetrically with cupferron after ion-exchange separation and obtained a mean value of 2.16%.

TITANIUM

Analysts Nos. 1, 2, 3, 6 and 9 determined titanium photometrically. Nos. 1, 2 and 6 with diantipyrylmethane and Nos. 3 and 9 with peroxide. Nos. 5, 7 and 10 used ICP-AES.

Analyst No. 2 also used ICP-AES and obtained a mean value of 0.87%. Analyst No. 10 also determined titanium photometrically with diantipyrylmethane and obtained a mean value of 0.88%.

TUNGSTEN

All analysts, except No. 1, determined tungsten using ICP-AES. No. 1 determined tungsten photometrically with thiocyanate using a synthetic calibration made by standard tungsten additions to a matched matrix containing molybdenum and titanium.

ZIRCONIUM

All analysts except No. 1 determined zirconium using ICP-AES. No. 1 determined zirconium photometrically with arsenazo III following a mercury cathode separation.

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NOTES ON METHODS USED

IRON

Analyst No. 1 determined iron titrimetrically with dichromate after reduction with stannous chloride. Nos. 2, 4, 6, 8, and 9 used FAAS and Nos. 5, 7 and 10 used ICP-AES. No. 3 determined iron photometrically with thiocyanate.

Analyst No. 2 also used ICP-AES and obtained a mean value of 1.51%. Analyst No. 10 also determined iron photometrically, after hydroxide separation, and obtained a mean value of 1.50%.

DESCRIPTION OF SAMPLE

*The BCS material is supplied in bottles of 100g chips graded 1700 – 250µm (10 – 60 mesh) for chemical analysis.

†The SS-CRM material is a Spectroscopic Standard and is supplied as discs 50mm dia x 13mm thick for spectroscopic analysis.

INTENDED USE & STABILITY

This sample is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is not possible and for establishing values for secondary reference materials.

It will remain stable provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened the lid should be secured immediately after use.

TRACEABILITY

The traceability of this CRM is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

This Certified Reference Material has been prepared in accordance with the recommendations specified in ISO Guides 30 to 35, available from the International Standards Organisation in Geneva.

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For BUREAU OF ANALYSED SAMPLES LTD.

P.D. RIDSDALE,
Chairman

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