PRELIMINARY EDITION

BUREAU OF ANALYSED SAMPLES LTD

UKAS REFERENCE MATERIALS
4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS/SS-CRM No. 351/1
IN 718 NICKEL ALLOY

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, AUSTRALIA, CANADA, THE NETHERLANDS AND THE UNITED STATES OF AMERICA, issued by the Bureau of Analysed Samples Ltd

ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Al	B	Co	Cu	N	Nb	Sn	Ti
1	0.0270	...	0.0581	0.0040	...	19.1025	3.0783	...	0.5581	0.0033	0.1455	0.0204	0.0068	5.2808	0.00033	0.9687
2	0.0240	0.0792	0.0595	0.0052	...	19.2748	3.0205	...	0.5600	0.0031	0.1565	0.0217	...	5.3828	0.00040	0.9020
3	0.0268	0.0843	0.0545	0.0050	...	19.2625	3.1150	53.1325	...	0.0034	0.1428	...	0.0066	5.2375	0.00035	0.9593
4	0.0262	...	...	...	0.00035	...	...	53.4913	...	...	...	...	0.0096	...	...	...
5	0.0256	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
6	0.0265	0.0845	0.0551	0.0035	0.00031	...	3.0060	...	0.5426	0.0039	0.1431	0.0214	0.0059	5.3158	0.00038	0.9306
7	0.0252	0.0744	0.0580	0.0041	0.00040	18.9825	...	...	0.5478	0.0036	0.1496	0.0200	0.0083	...	0.00014	0.9303
8	...	...	...	...	...	...	...	...	...	...	0.1337	...	0.0085	...	...	0.9000
9	0.0236	0.0883	0.0546	0.0053	0.00018	19.1748	3.0338	53.3743	0.5561	0.0028	0.1534	0.0224	0.0071	5.3400	0.00040	0.9155
10	0.0243	...	0.0537	...	...	...	...	...	...	...	0.1393	...	...	5.3193	...	0.9675
11	0.0256	...	...	...	0.00051	...	...	...	...	...	...	...	...	...	...	...
12	0.0265	0.0795	0.0538	0.0044	0.00045	19.1903	2.9955	53.3918	0.5500	0.0042	0.1418	0.0260	...	5.3378	0.00040	0.9708
13	0.0247	0.0678	0.0589	0.0045	0.00038	19.0460	3.0487	53.2300	0.5600	0.0033	0.1509	0.0233	0.0087	5.3236	0.00014	0.9460
14	0.0255	0.0828	0.0554	0.0043	0.00035	19.0750	3.0400	53.4750	0.5553	...	0.1363	...	...	5.2875	0.00041	0.9273
M_M	**0.0255**	**0.0801**	**0.0562**	**0.0045**	**0.00037**	**19.1386**	**3.0422**	**53.3492**	**0.5537**	**0.0035**	**0.1448**	**0.0222**	**0.0077**	**5.3139**	**0.00033**	**0.9380**
s_M	0.0011	0.0066	0.0023	0.0006	0.00010	0.1044	0.0391	0.1412	0.0064	0.0005	0.0072	0.0021	0.0013	0.0415	0.00011	0.0263
s_w	0.0007	0.0022	0.0006	0.0002	0.00005	0.0625	0.0245	0.1530	0.0057	0.0002	0.0053	0.0006	0.0004	0.0514	0.00004	0.0117

Analyst No.	V	W	Zr	Fe	Mg	Sb	Ta
1	0.0148	...	0.0017	17.2190	0.0011	0.00010	...
2	0.0181	0.0206	0.0020	17.1235	0.0014	0.00030	0.0028
3	0.0140	...	0.0015	...	0.0016	...	0.0032
4	...	...	...	...	...	...	...
5	...	...	...	...	...	...	...
6	0.0162	0.0235	0.0017	...	0.0015	0.00026	0.0038
7	0.0209	0.0195	0.0013	...	0.0012	0.00015	0.0048
8	...	...	...	...	...	...	...
9	0.0198	0.0202	0.0015	17.1865	0.0017	0.00030	0.0035
10	...	0.0205	0.0018	...	0.0013	...	...
11	...	...	...	...	...	...	...
12	...	...	...	17.1920	0.0015	0.00028	...
13	0.0217	0.0238	0.0017	17.1932	0.0021	0.00026	0.0022
14	0.0194	0.0182	0.0018	17.3000	0.0022	0.00028	0.0028
M_M	**0.0181**	**0.0209**	**0.0017**	**17.2024**	**0.0016**	**0.00024**	**0.0033**
s_M	0.0029	0.0021	0.0003	0.0574	0.0004	0.00008	0.0009
s_w	0.0004	0.0004	0.0002	0.0895	0.0002	0.00003	0.0002

As	*Ag*	*Bi*	*Ca*	*Pb*	*Se*	*Te*	*Tl*	*Zn*
0.000100	...	*<0.00005*	...	<0.0001	*<0.0001*	*<0.0001*	...	...
0.000825	<0.00002	*<0.00001*	*<0.001*	0.000018	*<0.0003*	*<0.00005*	*<0.00005*	*<0.0005*
<0.001	<0.0001	*<0.0001*	*<0.001*	<0.001	*<0.0001*	*<0.0001*	*<0.0001*	*<0.001*
...	...	...	...	...	...	...	...	...
0.000143	...	...	...	<0.00003	...	...	...	...
0.000625	0.000015	*<0.00001*	*0.00030*	<0.00002	*<0.00004*	*<0.00002*	*<0.00002*	*<0.0001*
0.000638	0.000004	*0.000001*	...	0.000014	*0.00002*	*0.000001*	*0.000001*	*0.00007*
<0.001	<0.01	*<0.01*	*<0.01*	<0.01	*<0.01*	*<0.01*	...	*<0.01*
0.000353	0.000015	*0.000013*	*0.00098*	0.000020	*<0.00001*	*0.00006*	*0.00006*	*0.000133*
...	...	...	...	...	...	...	...	...
0.000286	0.000007	*0.000007*	*0.00051*	0.000021	*<=0.00008*	*0.000027*	*0.000027*	*0.000086*
0.000625	<0.00001	*<0.00002*	*0.00028*	0.000025	*0.00004*	*<0.00002*	*<0.00002*	*0.0004*
0.000330	0.000002	*0.000011*	...	<0.00007	...	*0.00003*	*0.00003*	*0.00014*
0.000054	0.000003	*<0.000001*	*<0.001*	<0.00001	*<0.00004*	*<0.00002*	*<0.00002*	*<0.0002*

Values given in italics are for information only

Additional Information: Analyst No. 7 determined Cd and Ga by FAAS and found 0.000002% and 0.00154% respectively.

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

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	B	Co	Cu
Cv	**0.0255**	**0.080**	**0.0562**	**0.0045**	**0.00037**	**19.14**	**3.04**	**53.35**	**0.554**	**0.0035**	**0.145**	**0.0222**
C(95%)	0.0007	0.006	0.0016	0.0005	0.00009	0.09	0.04	0.15	0.006	0.0004	0.005	0.0019

	N	Nb	Sn	Ti	V	W	Zr	Fe	Mg	Sb	Ta
Cv	**0.0077**	**5.31**	**0.00033**	**0.938**	**0.0181**	**0.0209**	**0.0017**	**17.20**	**0.0016**	**0.00024**	**0.0033**
C(95%)	0.0011	0.04	0.00008	0.018	0.0024	0.0019	0.0002	0.07	0.0003	0.00007	0.0008

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.

BCS/SS-CRM No. 351/1
IN 718 NICKEL ALLOY
NOTES ON METHODS USED

CARBON

All Analysts determined carbon by combustion/infrared techniques, Nos. 2 and 5 and 12 following ASTM E1019, No. 10 ISO 7524-1985 and No. 14 ASTM E1915.

SILICON

Analysts Nos. 1, 3, 6, 9, 12, 13 and 14 determined silicon by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), Nos. 6 and 12 according to ASTM E2594-09

Analyst No. 2 used Inductively Couple Plasma-Mass Spectrometry (ICP-MS) whilst No. 7 determined silicon gravimetrically after dehydration with perchloric acid.

MANGANESE

Analysts Nos. 1, 3, 6, 9, 10, 12 13 and 14 determined manganese by ICP-OES. Analysts No. 6, 10 and 12 followed the method in ASTM E2594-09.

Analyst No. 2 used ICP-MS and No. 7 Flame Atomic Absorption Spectrometry (FAAS).

PHOSPHORUS

Analysts Nos. 1, 3, 6, 7 and 9 determined phosphorus by ICP-OES. Nos. 2, 13 and 14 used ICP-MS and No. 12 used a photometric method in which the molybdenum blue complex was extracted with 2-methylpropan-1-ol.

SULPHUR

All Analysts determined sulphur by combustion/infrared techniques; No. 12 following ASTM E1019 and No. 14 ASTM E1915.

CHROMIUM

Analysts Nos.1, 2, 9, 13 and 14 determined chromium by ICP-OES, No. 2 according to ASTM E 1479. Analysts Nos. 3, 7 and 12 used a potentiometric titration with Fe(II) according to ASTM E354.

MOLYBDENUM

Analysts Nos.1, 2, 3, 6, 9, 13 and 14 determined molybdenum by ICP-OES. Analyst No. 2 used the method in ASTM E 1479.

Analyst No. 12 determined molybdenum gravimetrically with benzoin oxime after separation using ion-exchange.

NICKEL

Analyst No. 3 determined nickel gravimetrically according to ISO 6352.

Analyst No. 4 separated nickel as the dimethylglyoxime complex and titrated nickel with EDTA using murexide. No. 12 also separated nickel with dimethylglyoxime but used the gravimetric method given in ASTM E354.

The remaining Analysts used ICP-OES.

ALUMINIUM

All Analysts determined aluminium by ICP-OES. Analyst No. 2 followed ASTM E 1479 whilst Nos. 6 and 12 followed ASTM 2594-09.

BORON

All Analysts, except for No. 2, determined boron by ICP-OES, Analyst No. 1 following ASTM 2594-09.

Analyst No. 2 used ICP-MS.

COBALT

All Analysts, except for No. 2, determined cobalt by ICP-OES, Analysts Nos. 10 and 12 following ASTM E2594-09.

Analyst No. 2 used ICP-MS.

COPPER

Analysts Nos. 1, 3, 7, 12 and 13 determined copper by ICP-OES, No. 12 using the method in ASTM 2594-09.

Analysts Nos. 2 and 6 used ICP-MS whilst Analyst No. 9 used FAAS.

NITROGEN

All Analysts determined nitrogen by thermal conductivity after decomposition in a graphite crucible; No. 6 following ASTM E 1019.

NIOBIUM

All Analysts except No. 12 determined niobium by ICP-MS. Analyst No. 2 followed ASTM E 1479 and No. 10 ASTM E2594-09.

Analyst No. 12 used a gravimetric method, precipitating the niobium with cupferron after extraction by ion exchange according to ASTM E1473.

TIN

Analysts Nos. 1, 9 and 12 determined tin by Electrothermal Atomic Absorption Spectrometry (ETAAS); No. 12 used a method based on ASTM 1184/ASTM 1770.

Analysts Nos. 2, 6, 13 and 14 used ICP-MS whilst No. 3 used ICP-OES and No. 7 FAAS.

Analyst No. 11 reported finding $\leq 0.00005\%$ using hollow cathode optical emission spectroscopy.

TITANIUM

All Analysts except No. 12 determined titanium by ICP-OES, .Analyst No 2 following ASTM E1479, and Nos. 6 and 10 ASTM E2594-09.

No. 12 used a photometric method with hydrogen peroxide after separation of titanium by ion exchange.

BCS/SS-CRM No. 351/1
IN 718 NICKEL ALLOY
NOTES ON METHODS USED

VANADIUM

Analysts Nos. 1, 3, 7, 9, 13 and 14 determined vanadium by ICP-OES whilst Analysts Nos. 2 and 6 used ICP-MS.

TUNGSTEN

Analysts Nos. 7, 9 and 10 determined tungsten by ICP-OES; No. 10 following ASTM E2594-09. The remaining analysts used ICP-MS.

ZIRCONIUM

All Analysts except for Nos. 2, 3 and 14 determined zirconium by ICP-OES. The other Analysts used ICP-MS.

IRON

All Analysts except for No. 12 determined iron by ICP-OES; No. 2 following ASTM E 1479.
Analyst No. 12 titrated Fe(III) with $K_2Cr_2O_7$ after a double separation with NH_4OH according to ASTM E354.

MAGNESIUM

Analysts Nos. 1, 3 and 6 determined magnesium by ICP-OES.
Analysts Nos. 2, 13 and 14 used ICP-MS.
Analysts Nos. 7, 9, 10 and 12 used FAAS; No. 10 following ASTM E2594-09 and No. 12 a method based on ASTM E1835.

Analyst No. 11 determined magnesium by hollow cathode optical emission spectroscopy and reported a value of 0.0010%

ANTIMONY

Analysts Nos. 1, 9 and 12 used ETAAS. No. 12 used a method based on ASTM 1184/ASTM 1770.
Analysts Nos. 2, 6, 13 and 14 used ICP-MS.
Analyst No. 7 used FAAS.

Analyst No. 11 determined antimony by hollow cathode optical emission spectroscopy and reported a value of 0.00013%

TANTALUM

Analysts Nos. 2, 3, 13 and 14 determined tantalum by ICP-MS. The other three Analysts used ICP-OES.

ARSENIC

Analysts Nos. 1, 5, 6, 9 and 12 determined arsenic by ETAAS; No.5 according to ASTM 1184 and No. 12 following a method based on ASTM E1184/E1770.
Analysts Nos. 2, 13 and 14 used ICP-MS.
Analysts Nos. 3 and 8 used ICP-OES.
Analyst No. 7 used FAAS.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

SILVER

Analysts Nos. 2, 6, 13 and 14 determined silver by ICP-MS.
Analysts Nos. 3 and 8 used ICP-OES.
Analyst No. 7 used FAAS and Nos. 9 and 12 ETAAS. No. 12 used a method based on ASTM 1184/ASTM 1770.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

BISMUTH

Analysts Nos. 1, 9 and 12 determined bismuth by ETAAS. No. 12 used a method based on ASTM 1184/ASTM 1770.
Analysts Nos. 2, 3, 6, 13 and 14 used ICP-MS
Analyst No. 7 used FAAS and No. 8 ICP-OES.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

CALCIUM

Analyst No. 2 determined calcium by ICP-MS.
Analysts Nos. 3, 6, 8 and 14 used ICP-OES.
Analysts No. 9 and 12 used FAAS; No. 12 used a method based on ASTM 1835.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

LEAD

Analyst Nos. 1, 5, 9 and 12 determined lead by ETAAS; No. 5 following ASTM 1184 and No. 12 using a method based on ASTM 1184/ASTM 1770.
Analyst Nos. 2, 3, 6, 13 and 14 used ICP-MS.
Analyst No. 7 used FAAS.
Analyst No. 8 used ICP-OES.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

SELENIUM

Analysts Nos. 1, 2, 6, 9 and 12 determined selenium by ETAAS. No. 2 followed ASTM E 1479
Analysts Nos. 3 and 14 used ICP-MS; No. 7 used FAAS and No. 8 ICP-OES.

TELLURIUM

Analysts Nos. 1, 2, 6, 9 and 12 determined tellurium by ETAAS. No. 2 followed ASTM E 1479and No. 12 ASTM 1184/ASTM 1770.
Analysts Nos. 3, 13 and 14 used ICP-MS; No. 7 used FAAS and No. 8 ICP-OES.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

THALLIUM

Analysts Nos. 2, 3, 6, 13 and 14 determined thallium by ICP-MS. No. 2 followed ASTM E 1479
Analyst No. 7 used FAAS and Nos. 9 and 12 ETAAS.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

ZINC

Analysts Nos. 2, 3, 13 and 14 determined zinc by ICP-MS. No. 2 followed ASTM E 1479
Analyst Nos.6, 7, 9 and 12 used FAAS, No. 12 used a method based on ASTM 1835. Nos. 8 and 10 used ICP-OES.
Analyst No. 11 used hollow cathode optical emission spectroscopy.

BCS/SS-CRM No. 351/1
IN 718 NICKEL ALLOY

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

1	THOMAS, B. (*Miss), BSc (Hons), MFSSoc* & COFFEY, R., *BSc (Hons)*	Exova, Middlesbrough.
2	SOMRACK, L.,	NSL Analytical Services, Cleveland, Ohio, USA.
3	STEEN-VORSTER, A	Laboratory Services International, Rotterdam, The Netherlands.

ANALYSTS representing MANUFACTURERS and USERS

4	BLACKWELL, M.,	ATI Allvac, Sheffield.
5	BOWMAN, M.,	Western Australia Speciality Alloys, Canning Vale, W Australia, Australia.
6	COLE, P.,	ATI Allvac, Monroe, North Carolina, USA.
7	FARLEY, E.,	Alcoa Howmet Ltd., Exeter.
8	HAITOS, A.,	Vale Inco Ltd, Port Colborne, Ontario, Canada.
9	JORDAN, J.L.,	SMC-Huntington Alloys, Huntington, West Virginia, USA.
10	LEROY, T,	Vale Inco, Mississauga, Ontario, Canada.
11	PARKER, J.,	Firth Rixson Plc, Glossop.
12	POLINKO, C.,	Carpenter Technologies, Reading, Pennsylvania, USA.
13	SCRIMSHIRE, P.,	IncoTest, Hereford.
14	WANG R.,	Vale Inco Ltd., Copper Cliff, Ontario, Canada.

DESCRIPTION OF SAMPLE

The BCS material is supplied in bottles of 100g chips nominally graded 1700 – 250µm (10 – 60 mesh) for chemical analysis.
The SS material is provided as discs approximately 44mm in diameter.

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 other 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. If the contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

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.

MEASUREMENT UNCERTAINTY

In establishing the measurement uncertainty of a Certified Reference Material four factors have to be considered: uncertainty arising from the analysis of the material, from the homogeneity examination of the material, from the long term storage of the material and from the transportation of the material. In the case of a nickel alloy the contributions from the long term storage and transportation of the CRM will be negligible.

In this particular case the homogeneity contribution is also negligible and therefore the measurement uncertainty has been achieved using the equation given at the bottom of page 1.

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 methods used 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.

All but one of the participating laboratories were accredited to ISO/IEC 17025 at the time of the analysis, although not necessarily for all of the constituents determined and not necessarily for the analysis of nickel alloys. It has been established statistically that there is no difference between the results of the accredited and the non-accredited laboratories.

Bureau of Analysed Samples Ltd is the Reference Material Producer as defined in ISO Guide 34:2009 section 3.1 and is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2009 and 35:2006.

Bureau of Analysed Samples Ltd is a UKAS Accredited Reference Material Producer No 4004.

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.

For BUREAU OF ANALYSED SAMPLES LTD.
NEWHAM HALL
NEWBY
MIDDLESBROUGH
ENGLAND
TS8 9EA
Email: enquiries@basrid.co.uk
Website: www.basrid.co.uk
R P MEERES,
Managing Director

Preliminary Edition …. .... ..March 2012