MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS/SS-CRM No. 387/1

NIMONIC 901 ALLOY

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

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Al	B	Co	Cu	Ti	Fe	Sb	*Pb*
1	0.033	0.067	0.027	0.0036	0.0029	11.28	5.75	41.3	0.22	0.017	0.016	0.0072	3.01	38.3	0.0001	*0.00002*
2	0.035	0.074	0.021	...	...	11.37	5.76	41.0	0.24	...	...	0.0074	2.91	38.7	...	...
3	0.031	0.075	...	0.0042	0.0027	11.46	5.89	41.5	0.22	0.015	0.020	0.0085	3.01	...	0.0002	*0.00004*
4	0.032	...	0.024	0.0036	0.0025	11.39	5.86	41.1	0.24	0.016	0.019	0.0070	...	...	...	...
5	0.033	0.055	0.024	0.0033	0.0024	11.27	5.77	41.3	0.26	0.018	0.021	0.0089	2.98	38.6	...	...
6	0.036	0.050	0.024	0.0025	0.0036	11.29	5.87	41.3	0.23	0.018	0.021	0.0068	3.01	38.2	0.0003	*0.00003*
7	0.034	0.046	0.030	0.0030	0.0030	11.33	5.85	41.2	0.24	0.016	0.024	0.0081	3.05	38.2	0.0005	*0.00001*
8	0.033	0.050	0.024	0.0032	0.0026	11.40	5.88	41.2	0.24	0.017	0.016	0.0070	3.00	38.2	0.0003	*0.00004*
M_M	**0.033**	**0.06**	**0.025**	**0.0033**	**0.0028**	**11.35**	**5.83**	**41.2**	**0.24**	**0.017**	**0.020**	**0.0076**	**3.00**	**38.4**	**0.0003**	*0.00003*
s_M	0.002	0.02	0.003	0.0006	0.0005	0.07	0.06	0.2	0.02	0.002	0.003	0.0008	0.05	0.3	0.0002	...

The above figures are those which each Analyst has decided upon after careful verification.
Figures in bold type certified, figures in small italic type only approximate.

M_M: Mean of the laboratory mean values. s_M: standard deviation of the laboratory mean values.

Approximate values for information: Ag ≤*0.00002*%, Bi <*0.0001*%, Nb *0.006*%

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	B	Co	Cu	Ti	Fe	Sb
Cv	**0.033**	**0.06**	**0.025**	**0.0033**	**0.0028**	**11.35**	**5.83**	**41.2**	**0.24**	**0.017**	**0.020**	**0.0076**	**3.00**	**38.4**	**0.0003**
C(95%)	0.002	0.02	0.003	0.0005	0.0004	0.06	0.05	0.2	0.02	0.002	0.003	0.0007	0.04	0.3	0.0002

The half width confidence interval **C**(95%) is an expression of the uncertainty of the certified value, where **C**(95%) = $\frac{t \times s_M}{\sqrt{n}}$ and "**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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(The current edition is available at www.basrid.co.uk)

BCS/SS-CRM 387/1 NIMONIC 901 ALLOY

NOTES ON METHODS USED

CARBON

Analysts Nos. 1 and 2 determined carbon by combustion and non-aqueous titration. The other Analysts used high frequency combustion and infrared absorption.

SILICON

Analyst No. I determined silicon photometrically as molybdenum blue. Nos. 2, 5, 6 and 7 determined silicon gravimetrically with perchloric acid. Nos. 3 and 8 used Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES)

MANGANESE

Analyst No. 1 determined manganese photometrically after oxidation with periodate. Nos. 2, 4, 5, 6, 7 and 8 used Flame Atomic Absorption Spectrometry (FAAS).

PHOSPHORUS

All Analysts, except No. 3, determined phosphorus photometrically, No. 1 as molybdenum blue after separation as ferric phosphate, the others as phosphovanadomolybdate. No. 6 removed chromium as chromylchloride. No. 3 used ICP-OES.

SULPHUR

All Analysts, except No. 1, determined sulphur by high frequency combustion and infrared absorption. No. 1 determined sulphur gravimetrically after chromatographic separation on alumina.

CHROMIUM

All Analysts, except Nos. 3 and 8, determined chromium titrimetrically after oxidation with persulphate. Nos. 3 and 8 used ICP-OES.

Analyst No. 8 also determined chromium titrimetrically and obtained a mean value of 11.34%.

MOLYBDENUM

Analysts Nos. 1, 2, 5 and 7 determined molybdenum photometrically as the oxythiocyanate, all except No. 2 after extraction of the complex. Nos. 3 and 8 used ICP-OES. Nos. 4 and 6 used FAAS.

Analyst No. 8 also determined molybdenum photometrically and obtained a mean value of 5.86%.

NICKEL

Analysts Nos. 1, 2, 4, 5 and 7 determined nickel titrimetrically after separation with dimethylglyoxime. Nos. 1 and 2 dissolved the precipitate in dilute sulphuric acid, boiled with an excess of ferric sulphate and titrated the ferrous salt produced with dichromate (Analoid Method No. 62). Nos. 4, 5 and 7 titrated with EDTA. Nos. 3 and 8 used ICP-OES. No. 6 determined nickel gravimetrically.

Analyst No. 8 also determined nickel cyanometrically and obtained a mean value of 41.0%.

ALUMINIUM

Analysts Nos. 1, 2, 4, 5, 6 and 7 determined aluminium using FAAS. Nos. 3 and 8 used ICP-OES.

BORON

Analysts Nos. 1, 4, 5, 6, 7 and 8 determined boron photometrically, Nos. 1, 4, 5 and 8 with curcumin and Nos. 6 and 7 with dianthrimide. No. 3 used ICP-OES.

Analyst No. 8 also determined boron by ICP-OES and obtained a mean value of 0.017%.

COBALT

Analyst No. 1 determined cobalt photometrically with nitroso-R-salt. The other Analysts, except No. 3, used FAAS. No. 3 used ICP-OES.

Analyst No. 1 also used FAAS and obtained a mean value of 0.015%.

COPPER

Analysts Nos. 1, 2, 4, 6 and 7 determined copper using FAAS. Nos. 3, 5 and 8 used ICP-OES.

TITANIUM

Analysts Nos. 1, 2, 5 and 7 determined titanium photometrically, Nos. 1, 5 and 7 with diantipyrylmethane, No. 2 with peroxide. Nos. 3 and 8 used ICP-OES. No. 6 used FAAS.

Analyst No. 8 also determined titanium photometrically with diantipyrylmethane and obtained a mean value of 3.02%.

IRON

Analysts Nos. 1, 2, 5 and 6 determined iron titrimetrically with potassium dichromate, Nos. 1, 2 and 6 after reduction with stannous chloride, No. 5 after reduction with hydrogen sulphide. No. 7 used FAAS and No. 8 used ICP-OES.

Analyst No. 8 also determined iron titrimetrically and obtained a mean value of 38.1%.

ANTIMONY

Analysts Nos. 1, 6, 7 and 8 used electrothermal atomization atomic absorption spectrometry. No. 3 used ICP-OES.

LEAD

Analysts Nos. 1, 6, 7 and 8 used electrothermal atomization atomic absorption spectrometry. No. 3 used ICP-OES.

DESCRIPTION OF SAMPLE

British Chemical Standard, BCS-CRM 387/1, is sold in bottles containing 100g chips, predominantly graded 2000 – 250 µm (10 - 60 mesh) for chemical analysis. Spectroscopic Standard, SS-CRM 387/1, is sold as 41 mm diameter x 13 mm thick discs for spectroscopic analysis. The preparation of representative samples for chemical analysis and certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

INTENDED USE

BCS-CRM 387/1 is intended for uses such as the verification of the accuracy and repeatability 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 387/1 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 should be linished before use to remove any protective coating.

STABILITY

BCS-CRM 387/1 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. SS-CRM 387/1 will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surfaces).

TRACEABILITY

The characterisation of BCS-CRM 387/1 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 387/1 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 387/1) has been assessed on the bulk material using one way ANOVA and has been found to be acceptable. Homogeneity of the discs (SS-CRM 387/1) 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.

COMMUTABILITY

BCS-CRM 387/1 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 387/1 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. MEDEIKYTE,
Lab and Manufacturing Manager

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