MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 310/1

NIMONIC '90' ALLOY

BS 3075: NA 19

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

CO-OPERATING ANALYSTS

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	Cr	Ni	Al	Co	Ti	Fe
1	0.065	0.46	0.35	19.47	58.7	1.09	17.20	2.41	0.27
2	0.067	0.47	0.35	19.52	58.6	1.10	17.08	2.45	0.24
3	0.069	0.45	0.35	19.45	58.6	1.04	17.08	2.41	0.25
4	0.068	0.47	0.35	19.44	58.6	1.05	17.10	2.47	0.27
5	0.069	0.49	0.37	19.37	58.7	1.02	16.88	2.43	0.24
6	0.070	0.44	0.34	19.39	58.6	1.07	16.91	2.39	0.25
7	0.070	0.44	0.36	19.37	58.5	1.06	16.90	2.46	0.24
M_M	**0.068**	**0.46**	**0.35**	**19.43**	**58.6**	**1.06**	**17.0**	**2.43**	**0.25**
s_M	0.002	0.02	0.01	0.06	0.1	0.03	0.1	0.03	0.02

Figures in bold type certified, figures in small italic type only approximate

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

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

CERTIFIED VALUES (Cv)

mass content in %.

	C	Si	Mn	Cr	Ni	Al	Co	Ti	Fe
Cv	**0.068**	**0.46**	**0.35**	**19.43**	**58.6**	**1.06**	**17.0**	**2.43**	**0.25**
C(95%)	0.002	0.02	0.01	0.06	0.1	0.03	0.1	0.03	0.02

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.

BCS-CRM No. 310/1 NIMONIC '90' ALLOY

NOTES ON METHODS USED

CARBON

Analysts No. 1, 2 and 7 determined carbon by non-aqueous titration (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399). Nos. 3, 4 and 5 used infra-red absorption methods. No. 6 determined carbon by electrical conductivity (Still, Dauncey and Chirnside, Analyst, 1954, **79**, 4; 1962, **87**, 530).

Analyst No. 2 also used a low-pressure and an infra-red absorption method which gave results of 0.065% and 0.069% respectively.

SILICON

All Analysts determined silicon gravimetrically after dehydration with perchloric acid (Nos. 1, 2, 5 and 7), with perchloric/sulphuric acid mixture (Nos. 3 and 4) or with sulphuric acid only (No. 6). Nos. 1 and 2 followed the procedure of the Standard method for iron and steel BS 1121: Part 10: 1967.

Analyst No. 2 also used a photometric molybdenum-blue method and found 0.48%.

MANGANESE

All Analysts except No. 5 determined manganese photometrically by oxidation with periodate (Nos. 1, 2, 4, 6 and 7) or persulphate (No. 3). Nos. 1 and 4 followed the procedure of the Standard method for iron and steel, BS 1121: Part 23: 1951. No. 5 oxidized with persulphate and titrated with arsenite.

CHROMIUM

All Analysts determined chromium titrimetrically by oxidation to the hexavalent state, followed by titration with standard ammonium ferrous sulphate solution. No. 1 used the Analoid method No. 37. Nos. 2 and 4 used the Standard method BS 1121: Part 13: 1954. No. 3 titrated potentiometrically.

NICKEL

Nickel was precipitated as the dimethylglyoxime complex in each case. Analysts Nos. 1 and 6 completed gravimetrically, Nos. 2 and 3 by cyanide titration and Nos. 4, 5 and 7 by titration with EDTA.

ALUMINIUM

Analysts Nos. 1, 2, 3, 5 and 6 determined aluminium either titrimetrically or gravimetrically after precipitation as the oxinate. Nos. 1, 3 and 5 separated interfering elements by electrolysis using a mercury-cathode and by precipitation with cupferron. No. 2 carried out mercury-cathode electrolysis and separated remaining interfering elements by precipitation first with ammonia then cupferron. No. 6 carried out mercury-cathode electrolysis, separated Group III hydroxides with ammonia and then separated titania from the ignited precipitate by fusion with sodium carbonate. In all cases any traces of interfering elements still remaining were completed in an ammoniacal tartrate-cyanide solution before precipitating aluminium with oxine. No. 2 followed the procedure of the Standard method for iron and steel BS 1121: Part 35: 1967. No. 4 carried out a mercury-cathode electrolysis, separated titanium and titrated aluminium potentiometrically with fluoride. No. 7 carried out a mercury-cathode electrolysis and determined aluminium by titrating to a fixed pH with 1% hydrochloric acid.

COBALT

Analysts Nos. 1, 2, 3, 4 and 6 determined cobalt by potentiometric titration with ferricyanide in a buffered citrate solution. Nos. 1 and 2 carried out preliminary separation of the cobalt with phenylthiohydantoic acid. No. 4 carried out a preliminary separation of the chromium as chromyl chloride. No. 6 carried out a preliminary separation of cobalt (together with manganese) by means of sodium hypobromite. Nos. 3, 4 and 6 made corrections for manganese which is titrated simultaneously. No. 5 determined cobalt gravimetrically by precipitation with 1-nitroso-2-naphthol and ignition to the oxide. No. 7 used a photometric method depending on the use of nitroso-R-salt.

Analyst No. 2 also carried out a direct potentiometric titration of cobalt after oxidation of the chromium and a further determination after removal of chromium as chromyl chloride; in both cases corrections were made for manganese. These methods gave results of 16.96% and 16.97% respectively.

TITANIUM

All Analysts except No. 5 determined titanium photometrically with hydrogen peroxide. Nos. 1, 2, 3 and 4 carried out direct determinations without any preliminary separations (see the Standard method for titanium in permanent magnet alloys BS 1121: Part 17B: 1951) whereas Nos. 6 and 7 made preliminary separations of the titanium with cupferron. No. 5 separated titanium from the bulk of the other alloying constituents by mercury-cathode electrolysis, then precipitated with cupferron and determined it gravimetrically by ignition to TiO_2.

Analyst No. 6 also determined titanium photometrically with peroxide after a mercury-cathode separation and found 2.36%.

IRON

Analysts Nos. 1, 4 and 5 determined iron titrimetrically. Nos. 1 and 4 separated iron by precipitation with ammonia, dissolved the hydroxide precipitate in hydrochloric acid, reduced with stannous chloride and titrated with dichromate. No. I followed the procedure of the ASTM method Designation E38-58. No. 5 titrated with permanganate. Nos. 2, 3, 6 and 7 used photometric methods. No. 2 precipitated iron and titanium with cupferron, fused the ignited precipitate with potassium bisulphate, extracted with hydrochloric acid and determined iron with 1, 10-phenanthroline. No. 3 separated iron as hydroxide and determined it with thiocyanate. Nos. 6 and 7 determined the iron with thioglycollic acid after separation with cupferron (No. 6) or *iso*-butyl acetate (No. 7).

DESCRIPTION OF SAMPLE

British Chemical Standard - bottles of 100g chips graded 1700 – 250 micron (10 - 60 mesh) for chemical analysis.

INTENDED USE & STABILITY

BCS-CRM 310/1 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. If the contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

TRACEABILITY

The traceability of BCS-CRM 310/1 to the SI is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

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For BUREAU OF ANALYSED SAMPLES LTD

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Preliminary Edition ..... February 1969
Main Edition ..... September 1969
Main Edition (revised with C(95%) and s_M values for each certified element and recalculated value for Cr) ..... August 2015