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Certificate of Analyses

B.C.S. No. 337 (S.S. No. 67*)
AUSTENITIC STAINLESS STEEL

To B.S. Specification 970: En 58F and En 58G

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

The standard bar was specially prepared by Edgar Allen & Co. Ltd., Sheffield.

ANALYSES

Analyst No.	C %	Si %	S %	P %	Mn %	Cr %	Ni %	Nb %	Ta %	Pb %	Co %	Cu %	V %
1	0·084	..	0·018	0·018	..	..	..	..	..	..	..	..	..
2	0·085	0·52	0·018	0·015	0·88	17·78	9·46	1·01	0·045	..	*0·034*	*0·10*	*0·02*
3	0·078	..	..	..	0·85	..	9·47	..	..	..	..	..	..
4	0·082	..	..	..	0·88	..	9·55	1·00	0·048	..	..	..	..
5	0·080	0·51	0·018	0·016	0·87	17·82	9·58	1·02	0·048	0·0011	..	..	..
6	..	0·52	..	..	0·87	..	9·48	..	..	..	..	..	..
7	..	..	..	..	..	17·79	9·52	..	..	0·0012	..	..	..
8	..	0·50	..	0·016	0·86	..	..	..	..	..	..	..	..
9	0·079	0·50	0·018	0·015	0·89	17·82	9·54	1·06	..	..	..	..	..
10	..	0·49	..	..	0·86	..	..	1·03	0·050	..	..	..	..
11	..	..	..	..	..	..	..	..	0·046	0·0011	..	..	..
12	..	0·49	..	..	..	..	9·55	..	..	..	..	..	..
13	0·082	..	..	0·016	0·86	17·81	..	1·00	0·049	0·0012	..	..	*0·02*
14	..	0·49	0·020	..	..	..	..	..	..	..	..	..	..
15	0·082	..	0·019	0·017	..	17·78	..	..	..	..	..	..	..
16	0·079	0·50	0·020	0·017	..	17·82	..	1·04	0·047	0·0012	..	..	..
17	..	..	0·017	..	..	17·84	..	1·01	..	..	..	..	..
Average	**0·081**	**0·50**	**0·018**	**0·016**	**0·87**	**17·8$_0$**	**9·52**	**1·02**	**0·048**	**0·0012**	..	..	..

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

The following aditional information has also been supplied:-
Admiralty Materials Laboratory, Holton Heath, Poole:- Ta 0·045% (neutron activation method).

**Due to slight segregation of certain elements an area $\frac{5}{16}$" in diameter in the centre of the spectrographic standards should be avoided for emission spectroscopy.*

[P.T.O.

CO-OPERATING ANALYSTS AND FIRMS

REFEREE ANALYSTS—INDEPENDENT

1. British Iron & Steel Research Association, (per P. H. Scholes, *A.Met.*), Sheffield.

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7. Bills, K. M., International Nickel Ltd., Birmingham.

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17. Taylor, L. N., Samuel Osborn & Co. Ltd., Sheffield.

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NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 2, 4 and 13 determined carbon by conductimetric methods. Nos. 1 and 13 used a balanced two-cell circuit, No. 2 used the G.E.C. method (Still, Dauncey and Chirnside, Analyst, 1954, **79**, 4) and No. 4 used a semi-micro method (Nall and Scholey, Metallurgia, 1961, **64**, 97). Nos. 3 and 9 used the Standard gravimetric method, B.S. 1121: Part 11: 1948, Nos. 5, 15 and 16 used a low-pressure method (Cook and Speight, Analyst, 1956, **81**, 144).

SILICON

All Analysts determined silicon gravimetrically by dehydration with perchloric acid according to the Standard method B.S. 1121: Part 10: 1948.

SULPHUR

Analysts Nos. 1, 15 and 16 determined sulphur by combustion. Nos. 1 and 15 used IRSID type combustion equipment (Pontet and Boulin, Chim. Anal., 1954, **38**, 98). No. 1 completed conductimetrically, Nos. 15 and 16 completed volumetrically. Nos. 2, 5, 9 and 17 determined sulphur gravimetrically; Nos. 2, 9 and 17 used the Standard method B.S. 1121: Part 1: 1966 in which the barium sulphate is precipitated in presence of the iron while No. 5 first separated sulphuric acid chromatographically on an alumina column (Nydahl, Anal. Chem., 1954, **26**, 580). No. 14 used both the Standard gravimetric method and a combustion method and reported the average of the results obtained.

Analyst No. 5 also used a combustion method and found 0·019%.

PHOSPHORUS

Analysts Nos. 1, 9 and 16 determined phosphorus by the Standard gravimetric method B.S. 1121: Part 9: 1948 in which the phosphorus is precipitated as ammonium phosphomolybdate and converted to lead molybdate. The other Analysts determined phosphorus colorimetrically as phosphovanadomolybdate; Nos. 2, 5, 13 and 15 extracted the coloured complex with *iso*-butyl methyl ketone according to the Draft B.S. method (in course of preparation, to be published as B.S. 1121: Part 45), No. 8 extracted with amyl alcohol.

MANGANESE

Analysts Nos. 2, 4, 5, 6, 8, 9, 10 and 13 determined manganese colorimetrically; Nos. 2, 4, 5, 8, 9 and 13 oxidized with periodate according to the Standard absorptiometric method B.S. 1121: Part 23: 1951, Nos. 6 and 10 oxidized with persulphate. No. 3 used the Standard volumetric method B.S. 1121: Part 16: 1951 which includes a zinc oxide separation.

Analyst No. 2 also used the British Standard volumetric method and found 0·85%. No. 3 also obtained a value of 0·88% by a colorimetric method.

CHROMIUM

All Analysts determined chromium volumetrically. No. 2 used the Analoid method No. 37 in which the hexavalent chromium is titrated directly with ammonium ferrous sulphate solution. No. 13 used a similar method but added excess ferrous solution and back-titrated with potassium dichromate. The other Analysts used the Standard method B.S. 1121: Part 13: 1954 in which the final titration is carried out with ammonium ferrous sulphate/permanganate.

NICKEL

All Analysts determined nickel volumetrically after precipitation with dimethylglyoxime. No. 2 dissolved the precipitate in dilute sulphuric acid and boiled with excess ferric sulphate; the ferrous salt thus formed was titrated with dichromate (Analoid Method, No. 62). Nos. 3, 4, 5, 6 and 12 titrated cyanometrically according to the Standard method B.S. 1121: Part 37: 1961. Nos. 7 and 9 titrated with EDTA.

Analyst No. 4 also used an EDTA method and found 9·44%. No. 12 also used a gravimetric method (Claassen and Bastings, Z. anal Chem., 1959, **165**, 354) and found 9·55%.

NIOBIUM

All Analysts except No. 4 determined niobium gravimetrically after separation of niobium and tantalum oxides by hydrolysis from a reduced solution (BISRA Methods of Analysis Committee, J. Iron Steel Inst., 1957, **187**, 341). In all cases a deduction was made for the tantalum which was determined separately. No. 4 separated niobium plus tantalum by precipitation with cupferron and determined niobium colorimetrically with thiocyanate in presence of stannous chloride (Bacon and Milner, Anal. Chim. Acta, 1956, **15**, 129).

Analyst No. 4 also determined niobium gravimetrically by hydrolysis from a reduced solution; the result, after making a deduction for tantalum, was 1·02%.

TANTALUM

Analyst No. 2 separated niobium plus tantalum by hydrolysis from a reduced solution and determined tantalum colorimetrically in the mixed oxides by means of pyrogallol (Kidman, Darwent and White, Metallurgia, 1960, **62**, 125). Nos. 4, 5, 10, 11, 13 and 16 separated niobium plus tantalum by precipitation with cupferron (No. 4), precipitation with phenylarsonic acid (Nos. 5, 11 and 13) or by hydrolysis from a reduced solution (Nos. 10 and 16); the tantalum was then separated from the niobium by solvent extraction with *iso*-butyl methyl ketone and determined colorimetrically with pyrogallol (Kidman and White, Metallurgia, 1961, **64**, 153).

LEAD

All Analysts separated the bulk of the iron by solvent extraction with amyl acetate, extracted the lead together with any bismuth into chloroform as the diethyldithiocarbamate and preferentially stripped the lead from this extract with hydrochloric acid; the lead was finally determined colorimetrically as the dithizonate (method developed by the BISRA Study Group for determination of traces of lead in steel; to be published).

COBALT

Standard method B.S. 1121: Part 42: 1961.

COPPER

Colorimetric method using *bis*-cyclohexanone oxalyldihydrazone (Analoid method No. 65).

VANADIUM

Analyst No. 2 separated the vanadium by precipitation with cupferron and determined it volumetrically. No. 13 separated iron and chromium by mercury cathode electrolysis and determined vanadium colorimetrically by a phosphovanadotungstate method.

For BUREAU OF ANALYSED SAMPLES LTD.
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Managing Director,
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