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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS*/SS[§]-CRM No. 345

NICKEL ALLOY IN 100

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

ANALYSES – MAIN ELEMENTS

Mean values – mass content in %.

| Analyst No. | C | Cr | Mo | Al | B | Co | Ti [†] | V | Zr |
|----------------------|--------------|-------------|-------------|-------------|--------------|--------------|-----------------|-------------|--------------|
| 1 | 0.156 | 9.88 | 3.05 | 5.64 | 0.017 | 14.74 | 4.71 | 1.05 | 0.045 |
| 3 | ... | 10.06 | ... | ... | ... | ... | ... | 0.96 | ... |
| 4 | 0.162 | 10.00 | 3.00 | 5.57 | 0.020 | 14.67 | 4.79 | 0.98 | 0.044 |
| 5 | 0.153 | 9.93 | ... | ... | 0.019 | ... | ... | 1.01 | 0.044 |
| 8 | 0.143 | 9.88 | 3.03 | 5.54 | ... | 14.75 | 4.74 | 0.97 | ... |
| 13 | 0.152 | ... | ... | ... | ... | ... | ... | ... | ... |
| 17 | 0.152 | 9.96 | 2.97 | 5.57 | 0.019 | 14.68 | 4.74 | 1.00 | 0.044 |
| M_M | 0.153 | 9.95 | 3.01 | 5.58 | 0.019 | 14.71 | 4.74 | 1.00 | 0.044 |
| C(95%) | 0.006 | 0.07 | 0.07 | 0.07 | 0.002 | 0.07 | 0.05 | 0.03 | 0.002 |
| s _M | 0.006 | 0.07 | 0.04 | 0.04 | 0.001 | 0.04 | 0.03 | 0.03 | 0.001 |
| s _w | 0.002 | 0.04 | 0.03 | 0.07 | 0.001 | 0.05 | 0.02 | 0.03 | 0.004 |

[†]The certified value applies **ONLY** to the finely divided form. Examination of the disc samples has revealed some radial segregation with respect to titanium towards the centre of the discs.

ANALYSES – TRACE ELEMENTS

Mean values – mass content in µg/g.

| Analyst No | Pb | Bi | Ag | Se | Te | Tl | Sb | Cd | Ga | Sn | Zn | Mg | As | Ca |
|----------------------|-------------|----------------|----------------|----------------|----------------|----------------|--------------|----------------|------------|------------|----------------|------------|-----|-----|
| 1 | 0.24 | <0.05 | <0.05 | <0.25 | <0.05 | <0.05 | <1.2 | <0.05 | 8.5 | 3.8 | <0.5 | 4.6 | 1.9 | <5 |
| 2 | 0.21 | 0.13 | <0.1 | ... | ... | ... | <1 | <0.1 | 8.1 | ... | <0.2 | 5.0 | 3.0 | ... |
| 4 | 0.18 | 0.05 | 0.08 | <0.5 | 0.11 | 0.06 | ... | ... | ... | ... | ... | 6.8 | ... | ... |
| 6 | 0.2 | <0.1 | <0.2 | <0.5 | <0.1 | ... | <1 | <0.1 | 8.5 | 8.0 | 0.3 | 7.2 | ... | <1 |
| 7 | ... | ... | <0.1 | ... | ... | ... | ... | <0.1 | ... | ... | ... | 3.9 | ... | ... |
| 9 | ... | <0.1 | <0.1 | ... | <0.2 | ... | ... | ... | 7.8 | ... | ... | ... | ... | ... |
| 10 | ... | ... | ... | ... | ... | ... | <1 | ... | ... | 5.9 | <0.5 | 5.0 | ... | ... |
| 11 | 0.30 | <0.2 | <0.1 | <0.5 | <0.1 | <0.1 | ... | ... | ... | ... | ... | ... | ... | ... |
| 12 | 0.21 | <0.05 | <0.05 | <0.2 | <0.1 | <0.1 | ... | ... | ... | 4.5 | 0.3 | 4.2 | 2.0 | <1 |
| 13 | 0.18 | <0.2 | <0.05 | <0.5 | <0.2 | <0.05 | 1.5 | <0.02 | 8.5 | 4.7 | 0.3 | 6.7 | ... | ... |
| 14 | 0.22 | <0.1 | <0.03 | <0.5 | <0.2 | <0.1 | ... | <0.1 | ... | ... | <0.5 | ... | ... | ... |
| 15 | ... | ... | ... | ... | ... | ... | ... | ... | ... | 6.8 | ... | ... | ... | ... |
| 16 | ... | ... | <0.02 | ... | ... | ... | ... | ... | ... | ... | ... | 6.0 | ... | ... |
| 17 | 0.12 | <0.2 | ... | ... | <0.2 | <0.2 | ... | ... | 8.0 | ... | ... | ... | 1.4 | ... |
| M_M | 0.21 | <0.2 | <0.2 | <0.5 | <0.2 | <0.2 | <2 | <0.1 | 8.2 | 5.6 | <0.5 | 5.5 | ... | ... |
| C(95%) | 0.04 | ... | ... | ... | ... | ... | ... | ... | 0.3 | 1.7 | ... | 0.9 | ... | ... |
| s _M | 0.05 | ... | ... | ... | ... | ... | ... | ... | 0.3 | 1.6 | ... | 1.2 | ... | ... |
| s _w | 0.06 | ... | ... | ... | ... | ... | ... | ... | 1.5 | 1.2 | ... | 1.0 | ... | ... |

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. s_w: Intralaboratory standard deviation.

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.

DESCRIPTION OF SAMPLE

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

§Spectroscopic Standard – 38 mm diameter discs for spectroscopic analysis

CO-OPERATING ANALYSTS AND FIRMS

INDEPENDENT ANALYST

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GOVERNMENT DEPARTMENT

2. NICHOLLS, H.A., *C.Chem., F.R.S.C.*, Materials Quality Assurance Directorate, Bragg Laboratory, Sheffield.

ANALYSTS representing MAKERS and USERS

3. BARNETT, D., Brookside Metal Co. Ltd., Willenhall.
4. BATTY, G., Ross and Catherall Ltd., Sheffield.
5. BILLS, K.M., Inco-Europe, Birmingham.
6. CALDWELL, J.A., *B.Sc., C.Chem., F.R.S.C.*, Murex Ltd., Rainham.
7. DEMARTEAU, L., Fabrique Nationale S.A., Herstal, Belgium.
8. DOUGHTY, A., APV Paramount Ltd., Crawley.
9. EVANS, R.D., & ALLEN, R.S., Rolls-Royce Ltd, Derby & Bristol.
10. GRIER, D.A., Cameron Iron Works, Livingston, Edinburgh.
11. HAIRE, R.J., Howmet Turbine Components Corporation, New Jersey, USA.
12. JACOBS, G.L., & NEWMAN, J.A., Cameron Iron Works Inc., Texas, USA.
13. MURPHY, J.P., Glossop Superalloys, Glossop.
14. OTT, W.L., Falconbridge Nickel Mines Ltd., Thornhill, Ontario, Canada.
15. RANSON, L., *B.Sc., M.Sc., Ph.D.*, London and Scandinavian Metallurgical Co. Ltd., Rotherham.
16. SHAW, D.H., Howmet Alloys International, Exeter.
17. THORNTON, K.A., *B.A.*, Wiggin Alloys Ltd., Hereford.

**BCS/SS-CRM No. 345
NICKEL ALLOY IN 100**

NOTES ON METHODS USED

MAIN ELEMENTS

CARBON

Analysts Nos. 1 and 8 determined carbon by combustion and non-aqueous titration according to the British Standard Carbon Method 4⁴. Nos. 4, 5, 13 and 17 used high frequency combustion with infrared absorption.

CHROMIUM

All analysts determined chromium by titration with ammonium ferrous sulphate after oxidation with persulphate/silver nitrate. Nos. 1, 3 and 8 followed the procedure of the British Standard Chromium Method 1⁴. No. 17 titrated potentiometrically.

Analyst No.8 also determined chromium by FAAS and found 9.90%.

MOLYBDENUM

Analysts Nos. 1, 4 and 17 determined molybdenum photometrically as oxythiocyanate. No. 1 followed the British Standard Molybdenum Method 1⁴ which involves extraction into n-butyl acetate. Analyst No.8 used FAAS.

ALUMINIUM

Analyst No. 1 separated aluminium using mercury cathode electrolysis and cupferron precipitation. The determination was completed by using sodium fluoride to release an amount of EDTA equivalent to the aluminium which was then titrated with zinc solution (Analoid Method No. 71). Analysts Nos. 8 and 17 used FAAS. No. 4 used a gravimetric method involving precipitation with 8-hydroxyquinoline after separation of interfering elements.

BORON

Analysts Nos. 1 and 5 separated boron by distillation as methyl borate and completed photometrically with carmine. Nos. 4 and 17 used dianthrime photometric methods without prior separation.

COBALT

Analysts Nos. 1, 4, and 8 determined cobalt photometrically with nitroso-R salt. No. 17 used a ferricyanide titrimetric method with a potentiometric end point.

Analyst No.8 also determined cobalt by FAAS and found 14.81%.

TITANIUM

Analysts Nos. 1, 4 and 17 determined titanium photometrically with hydrogen peroxide. Nos. 1 and 4 carried out a preliminary separation with cupferron. No.8 used FAAS.

VANADIUM

Analysts Nos. 1, 3 and 17 determined vanadium titrimetrically with ammonium ferrous sulphate. No. 1 according to the Analoid Method No. 34 and No. 3 according to the British Standard Vanadium Method 1⁴. No. 4 used a phosphovanadotungstate photometric method. Nos. 5 and 8 used FAAS.

ZIRCONIUM

Analysts Nos. 1, 4, 5 and 17 determined zirconium photometrically after separation by mercury cathode electrolysis. Nos. 1, 5 and 17 used arsenazo III and No. 4 xylenol orange.

TRACE ELEMENTS

LEAD

Analyst No.1 determined lead by FAAS after extraction into 4-methylpentan-2-one/TOPO from an iodide solution¹. No.2 separated lead by anion exchange and completed by ETA. Analysts Nos. 4, 6, 11, 12, 13 and 14 used ETA directly on an acid solution of the alloy prepared, in most cases, by dissolving in hydrofluoric/nitric acids and evaporating off most of the excess acids². Analyst No. 17 extracted lead into 4-methylpentan-2-one from an iodide solution and measured by square wave polarography after converting to an aqueous medium.

Analyst No. 17 also determined lead by FAAS after TOPO extraction and found 0.2µg/g.

BISMUTH

All Analysts except Nos. 9 and 17 used ETA. No. 1 separated bismuth by extraction into 4-methylpentan-2-one/TOPO from an iodide solution¹ and back extracted into nitric acid, No.2 used anion exchange and Nos. 4, 11, 12, 13 and 14 used the direct method described for lead. No. 6 extracted with 4-methylpentan-2-one/TOPO. Analyst No.9 used hydride generation FAAS. No. 17 used square wave polarography after extraction with iso-octyl thioglycollate.

Analyst No. 6 also determined bismuth by direct solution ETA and found 0.1µg/g. No. 17 also used FAAS after TOPO extraction and found 0.2µg/g.

SILVER

Analysts Nos. 1, 4, 6, 9, 11, 12, 13, 14 and 16 determined silver by direct ETA on an acid solution of the alloy, as described under lead². No.2 used ETA after separation by anion exchange. No. 7 used direct FAAS.

SELENIUM

All analysts determined selenium by ETA on an acid solution of the alloy prepared, in most cases, by dissolving in hydrofluoric/nitric acids and evaporating off most of the excess acids².

TELLURIUM

Analyst No. 1 separated tellurium by extraction as described under lead¹ and completed by ETA. Nos. 4, 11, 12, 13 and 14, used ETA directly on acid solutions². No. 6 first extracted with 4-methylpentan-2-one/TOPO and completed by ETA. Analyst No. 9 used hydride generation FAAS. No. 17 used square wave polarography after extraction with iso-octyl thioglycollate.

THALLIUM

Analyst No. 1 separated thallium by extraction as described under lead¹ and completed by ETA. Nos. 4, 11, 12, 13, and 14 used ETA directly on acid solutions²; No. 14 added sulphuric acid as a precaution against loss of thallium on pyrolysis. No. 17 extracted with 4-methylpentan-2-one/TOPO and completed by FAAS.

Analyst No. 16 determined thallium by direct ETA on the solid alloy and found 0.1µg/g.

ANTIMONY

Analysts Nos. 1 and 10 determined antimony by FAAS after extraction into 4-methylpentan-2-one/TOPO from an iodide solution¹. Nos. 2 and 13 used ETA directly on acid solutions². No. 6 first extracted with 4-methylpentan-2-one/TOPO and completed by ETA.

Analysts Nos. 1 and 6 also determined antimony by direct ETA on an acid solution and found 1.2 1.0µg/g respectively.

CADMIUM

Analysts Nos. 1, 6, 7, 13 and 14 determined cadmium by ETA directly on acid solutions². No. 2 used ETA after separation by anion exchange.

GALLIUM

Analyst No. 1 determined gallium by FAAS and No. 13 by ETA after extraction with 4-methylpentan-2-one/TOPO from a 7 molar hydrochloric acid solution. No. 2 extracted gallium as chloride with diethyl ether, removed molybdenum with α -benzoin oxime and evaluated by FAES. Analyst No. 6 used a photometric method with brilliant green. Nos. 9 and 17 used ETA directly on an acid solution.

TIN

Analysts Nos. 1, 10 and 15 determined tin by FAAS after extraction with 4-methylpentan-2-one/TOPO from an iodide solution¹. The remaining analysts used ETA directly on acid solutions².

Analyst No. 6 also determined tin by ETA after extraction into 4-methylpentan-2-one/TOPO and found 8.5µg/g.

ZINC

Analysts Nos. 1, 6, 10, 12 and 14 determined zinc by FAAS without separation. Nos. 2 and 13 used ETA directly on acid solutions².

MAGNESIUM

Analysts Nos. 1, 2, 4, 6, 7, 10, 12 and 16 determined magnesium by FAAS without separation. No. 13 used ETA directly on an acid solution.

ARSENIC

Analyst No. 1 separated arsenic by extraction as the iodide into chloroform³, back extracted into water and acidified with nitric acid. The resulting solution was evaluated using ETA. No. 17 used a similar extraction procedure, using chloride instead of iodide, and completed photometrically as molybdenum blue. Analyst No. 2 used a photometric method involving the evolution of arsine and absorption in silver diethyldithiocarbamate in chloroform. No. 12 used ETA directly on an acid solution².

Analyst No. 1 also used a silver diethyldithiocarbamate photometric method and found 1.4µg/g.

CALCIUM

All analysts used direct FAAS.

References

1. Burke, Analyst 1972, **97**, 19 and Burke and Thornton, Analyst, 1974, **99**, 469
2. Welcher, Krieger and Marks, Anal. Chem., 1974, **46**, 1227
3. Fogg, Marriott and Thorburn-Burns, Analyst 1972, **97**, 657
4. Methods for Sampling and Analysis of Iron and Steel, 1970, British Standards Institution, London

Abbreviations

FAAS : Flame atomic absorption spectrometry
ETA : Electrothermal atomisation (atomic absorption spectrometry)
FAES : Flame atomic emission spectrometry
TOPO : Tri-n-octylphosphine oxide

Traceability: The majority of Analysts calibrated using pure metals, metal oxides or primary chemicals.

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MIDDLESBROUGH
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For BUREAU OF ANALYSED SAMPLES LTD.
P.D. RIDSDALE,
Chairman

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