



# BUREAU OF ANALYSED SAMPLES LTD.

Directors: -

P.D.RIDSDALE, *B.Sc., C.Eng., M.I.M., M.R.S.C.* (Chairman)R. P. MEERES, *B.A., (Oxon), M.R.S.C.* (Managing)G. C. FLINTOFT, *A.C.M.A.*

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

## CERTIFICATE OF ANALYSIS

### BCS - CRM No. 346

### 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 – TRACE ELEMENTS

Mean values – mass content in µg/g.

Analyst No	Pb	Bi	Ag	Se	Te	Tl	Sb	As	Cd	Ga	Sn	Zn	Mg	Ca	In
1	19.0	9.4	34.5	10.2	9.5	1.7	45.4	50.0	0.34	50.0	90	28.8	136	41	20
2	20.5	10.5	37.7	...	...	...	43.8	51.8	0.48	50.7	...	27.6	144	...	...
3	18.0	10.9	34.8	8.5	11.2	1.7	...	50.5	...	...	...	...	161	...	...
4	20.5	10.4	33.5	7.5	10.3	...	46.0	...	0.40	50.5	90	31.5	158	42	...
5	23.5	10.3	38.1	...	11.5	...	...	...	0.42	...	...	...	150	...	...
6	...	...	...	...	...	...	...	...	...	...	...	25.7	...	39	...
7	23.2	...	34.2	...	14.5	1.9	...	...	...	49.5	...	...	...	...	...
8	22.8	10.5	36.2	...	...	...	49.0	...	...	...	99	28.8	137	...	...
9	20.4	10.8	34.8	8.8	12.1	1.4	...	...	...	...	...	...	...	...	...
10	22.0	9.6	35.8	10.0	11.1	1.8	51.0	52.0	...	...	100	28.5	138	38	...
11	19.6	10.4	33.8	8.9	11.8	1.7	...	...	0.44	...	92	29.1	141	33	19
12	21.9	11.6	34.6	10.0	12.8	2.1	...	...	...	...	...	31.2	...	...	...
13	22.0	10.0	32.2	...	...	...	...	...	...	...	87	...	...	...	...
14	20.4	10.8	34.4	...	...	...	...	...	...	...	78	...	156	...	18
15	19.5	9.9	...	...	12.1	1.7	...	47.0	...	52.3	...	...	...	...	...
<b>M<sub>M</sub></b>	<b>21.0</b>	<b>10.4</b>	<b>35.0</b>	<b>9.1</b>	<b>11.7</b>	<b>1.8</b>	<b>47.0</b>	<b>50.3</b>	<b>0.42</b>	<b>50.6</b>	<b>91</b>	<b>28.9</b>	<b>147</b>	...	...
<i>s<sub>M</sub></i>	1.7	0.6	1.7	1.0	1.4	0.2	2.9	2.0	0.05	1.1	8	1.9	10	...	...
<i>s<sub>w</sub></i>	2.6	1.7	1.3	0.9	1.2	0.2	0.9	2.7	0.09	3.0	7	0.6	4	...	...

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

**M<sub>M</sub>**: Mean of the intralaboratory means. *s<sub>M</sub>*: standard deviation of the intralaboratory means. *s<sub>w</sub>*: Intralaboratory standard deviation.

Values given above in italic type are for information only

#### CERTIFIED VALUES

mass content in µg/g

Analyst No	Pb	Bi	Ag	Se	Te	Tl	Sb	As	Cd	Ga	Sn	Zn	Mg
<b>M<sub>M</sub></b>	<b>21.0</b>	<b>10.4</b>	<b>35.0</b>	<b>9.1</b>	<b>11.7</b>	<b>1.8</b>	<b>47</b>	<b>50.3</b>	<b>0.42</b>	<b>50.6</b>	<b>91</b>	<b>28.9</b>	<b>147</b>
C(95%)	1.0	0.4	1.0	0.9	1.0	0.2	4	2.5	0.06	1.4	8	1.6	7

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.

The composition of this alloy is similar to that of BCS/SS – CRM 345, i.e. C 0.15%, Cr 10%, Mo 3%, Al, 5.5%, Co 15%, Ti 5%, V 1% and Ni 60%.

#### DESCRIPTION OF SAMPLE

This sample is only available in the form of chips graded 1700 - 250µm (10 – 60 mesh)

## CO-OPERATING ANALYSTS AND FIRMS

### INDEPENDENT ANALYST

1. PICKERING, J.L., *C.Chem., M.R.S.C.*, Ridsdale & Co Ltd., Middlesbrough.

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

**BCS - CRM No. 346**  
**NICKEL ALLOY IN 100**  
**NOTES ON METHODS USED**

**LEAD**

Analysts Nos. 1, 4, 5, 7, 9, 10, 12 and 14 determined lead by ETA AAS 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 acids<sup>1</sup>. Analysts Nos. 2 and 3 used FAAS without separation. Nos. 8, 11 and 13 extracted lead into 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup> and completed by FAAS on the organic extract. Analyst No. 15 extracted lead into 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup> and measured by square wave polarography after conversion to an aqueous medium.

Analysts No. 4 and 15 also determined lead by FAAS after extraction into 4-methylpentan-2-one/TOPO and found 21.0 and 19.8 µg/g respectively.

**BISMUTH**

Analysts Nos. 1, 5, 9, 10, 12 and 14 determined bismuth by ETA directly on an acid solution as described under Lead. No. 2 separated bismuth by anion exchange and completed by ETA. Nos. 3, 4, 8, 11 and 13 used FAAS after extraction into 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup>. Analyst No. 15 used square wave polarography for measurement after extraction with iso-octyl thioglycollate.

Analyst No. 4 also determined bismuth by ETA directly on an acid solution and found 10.0µg/g. No. 15 also used FAAS after 4-methylpentan-2-one/TOPO extraction and found 10.4µg/g.

**SILVER**

Analysts Nos. 1, 2, 3, 4, 5, 7 and 10 determined silver by FAAS without separation. Nos. 8 and 13 extracted with 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup> and completed by FAAS. Nos. 9, 11, 12 and 14 used ETA directly on an acid solution as described under Lead.

**SELENIUM**

All analysts determined selenium by ETA on acid solution of the alloy prepared, in most cases, by dissolving in hydrofluoric/nitric acids and evaporating off most of the excess acids<sup>1</sup>.

**TELLURIUM**

Analysts Nos. 1, 3, 5, 7, 9, 10 and 12 determined tellurium by ETA on an acid solution of the alloy<sup>1</sup>. Nos. 4 and 11 extracted with 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup> and completed by FAAS. No. 15 used square wave polarography for measurement after extraction with iso-octyl thioglycollate.

**THALLIUM**

Analysts Nos. 1, 11 and 15 extracted with 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup>. Nos. 1 and 15 completed by FAAS and No. 11 completed by ETA. Nos. 3, 9, 10 and 12 used ETA directly on acid solutions<sup>1</sup>.

**ANTIMONY**

Analysts Nos. 1, 4 and 8 determined antimony by FAAS after extraction in 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup>. Nos. 2 and 10 used ETA directly on acid solutions<sup>1</sup>.

Analyst No. 4 also determined antimony by ETA directly on an acid solution and found 45.0µg/g.

**ARSENIC**

Analysts Nos. 1 and 15 extracted arsenic as iodide<sup>3</sup> and chloride respectively into chloroform and completed photometrically as molybdenum blue. Nos. 2 and 10 used ETA directly on an acid solution<sup>1</sup>. Analyst No. 3 determined arsenic by hydride vapour FAAS.

Analyst No. 1 also determined arsenic by FAAS using an iodide extraction as described above, acidified with nitric acid and found 47.0µg/g. No. 2 also used a photometric method involving absorption of arsine in silver diethyldithiocarbamate/ephedrine in chloroform and found 55.5µg/g.

**CADMIUM**

Analysts Nos. 1, 4, 5 and 11 determined cadmium by ETA directly on acid solutions<sup>1</sup>. No. 2 used ETA after separation by anion exchange.

**GALLIUM**

Analyst No. 1 determined gallium by FAAS after extraction with 4-methylpentan-2-one/TOPO from a 7 molar hydrochloric acid solution. No. 2 extracted gallium as the chloride with diethyl ether, removed molybdenum with α-benzoin oxime and evaluated by FAAS. No. 4 used a photometric method with brilliant green. No. 7 used ETA directly on an acid solution. Analyst No. 15 determined gallium by ETA directly on an acid solution<sup>1</sup>.

**TIN**

Analysts Nos. 1, 8, 11, and 13 determined tin by FAAS after extraction with 4-methylpentan-2-one/TOPO from an iodide solution<sup>2</sup>. The remaining analysts used ETA directly on acid solutions<sup>1</sup>.

**ZINC**

All analysts determined zinc by FAAS directly.

## MAGNESIUM

All analysts determined magnesium by FAAS directly.

## CALCIUM

All analysts used direct FAAS.

## INDIUM

All analysts determined indium by ETA after extraction with 4-methylpentan-2-one/TOPO from a 7 molar hydrochloric acid solution, No.14 after digestion with hydrofluoric and nitric acids.

## References

1. Welcher, Krieger and Marks, Anal. Chem., 1974, **46**, 1227
2. Burke, Analyst 1972, **97**, 19 and Burke and Thornton, Analyst, 1974, **99**, 469
3. Fogg, Marriott and Thorburn-Burns, Analyst 1972, **97**, 657

## 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.

NEWHAM HALL  
MIDDLESBROUGH  
ENGLAND

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