MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

SPECTROSCOPIC STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
SS-CRM No. 346A
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.

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ANALYSES

Mean of 4 values - mass content in µg/g.

Analyst No.	Pb	Bi	Ag	Se	Te	Tl	Sb	As	Cd	Ga	Sn	Zn	Mg	*Ca*	*In*
1	20.0	9.2	42.5	6.7	8.2	1.7	45.5	51.2	0.34	47.9	92	28.0	118	*20*	*19*
2	22.0	9.0	44.5	...	...	...	42.0	51.8	0.38	49.2	...	27.4	126	...	...
3	20.6	10.6	41.2	6.0	8.0	...	...	50.2	...	...	...	...	139	...	...
4	20.0	10.4	40.5	4.9	9.8	...	49.0	...	0.35	48.5	95	31.6	143	*28*	...
5	25.0	10.2	45.0	...	8.4	2.3	...	...	0.40	...	...	...	134	...	...
6	...	...	...	...	...	...	...	...	...	...	...	27.3	...	...	...
7	24.8	12.5	41.5	...	...	1.9	...	...	...	51.0	...	...	...	...	...
8	23.0	10.6	43.0	...	...	...	48.0	...	...	...	102	28.0	123	...	...
9	21.1	10.6	41.2	5.0	9.4	...	...	...	...	...	...	...	...	...	...
10	22.5	9.0	42.9	6.5	9.2	1.7	47.0	51.8	...	...	102	28.0	122	*21*	...
11	19.9	10.4	43.3	4.8	10.8	1.7	38.8	...	...	...	89	29.4	123	*17*	*20*
12	23.2	11.8	43.2	6.3	10.2	2.1	...	...	0.40	...	...	30.9	...	...	...
13	25.8	9.2	...	...	...	...	...	...	...	...	81	...	...	...	...
14	21.2	10.6	41.0	...	...	...	...	...	...	...	90	...	140	...	*22*
15	21.4	9.8	...	...	9.4	1.7	...	47.1	...	51.6	...	...	...	...	...
M_M	**22.2**	**10.3**	**42.5**	**5.7**	**9.3**	**1.9**	**45.1**	**50.4**	**0.37**	**49.6**	**93**	**28.8**	**130**	*22*	*20*
s_M	2.0	1.1	1.5	0.9	1.0	0.3	4.0	2.0	0.03	1.6	8	1.7	10	...	...
s_W	3.0	1.2	1.9	0.6	1.2	0.3	2.1	2.6	0.03	2.1	4	1.6	7	...	...

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, s_W: Average within laboratory standard deviation.

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%.

CERTIFIED VALUES (Cv)

mass content in µg/g

	Pb	Bi	Ag	Se	Te	Tl	Sb	As	Cd	Ga	Sn	Zn	Mg
Cv	**22.2**	**10.3**	**42.5**	**5.7**	**9.3**	**1.9**	**45**	**50.4**	**0.37**	**49.6**	**93**	**28.8**	**130**
C(95%)	1.2	0.6	1.0	0.8	0.8	0.3	5	2.5	0.04	2.0	7	1.4	8

The half width confidence interval, **C**(95%), is an expression of the uncertainty of the certified value.

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.

DESCRIPTION OF SAMPLE

Spectroscopic Standard, SS-CRM 346A, is sold as 38mm diameter and 13mm thick discs for spectroscopic analysis.

The preparation of representative samples for the chemical analysis and certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

Date of this edition: October 2019.

(The current edition is available at www.basrid.co.uk)

SS-CRM 346A NICKEL ALLOY IN 100

NOTES ON METHODS USED

LEAD

Analysts Nos. 1, 4, 5, 7, 9, 10, 12 and 14 determined lead by Electrothermal Atomisation Atomic Absorption Spectrometry (**ETAAS**) 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[1]. Analysts Nos. 2 and 3 used Flame Atomic Absorption Spectrometry (**FAAS**) without separation. Nos. 8, 11 and 13 extracted lead into 4-methylpentan-2-one/Tri-n-octylphosphine oxide (**TOPO)** from an iodide solution[2] and completed by FAAS on the organic extract. Analyst No. 15 extracted lead into 4-methylpentan-2-one/TOPO from an iodide solution[2] and measured by square wave polarography after conversion to an aqueous medium.

Analysts Nos. 4 and 15 also determined lead by FAAS after extraction into 4-methylpentan-2-one/TOPO and found 21.2 and 20.9µg/g respectively.

BISMUTH

Analysts Nos. 1, 5, 7, 9, 10, 12 and 14 determined bismuth by ETAAS directly on an acid solution as described under Lead. No. 2 separated bismuth by anion exchange and completed by ETAAS. Nos. 3, 4, 8, 11 and 13 used FAAS after extraction into 4-methylpentan-2-one/TOPO from an iodide soluton[2]. Analyst No. 15 used square wave polarography for measurement after extraction with iso-octyl thioglycollate.

Analyst No. 4 also determined bismuth by ETAAS directly on an acid solution and found 11.0µg/g. No. 15 also used FAAS after 4-methylpentan-2-one/TOPO extraction and found 10.0µg/g.

SILVER

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

SELENIUM

All Analysts determined selenium by ETAAS on acid solution of the alloy prepared, in most cases, by dissolving in hydrofluoric/nitric acids and evaporating off most of the excess acids[1].

TELLURIUM

Analysts Nos. 1, 3, 5, 7, 9, 10 and 12 determined tellurium by ETAAS on an acid solution of the alloy[1]. Nos. 4 and 11 extracted with 4-methylpentan-2-one/TOPO from an iodide solution[2] 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[2]. Nos. 1 and 15 completed by FAAS and No. 11 completed by ETAAS. Nos. 5, 10 and 12 used ETAAS directly on acid solutions[1].

ANTIMONY

Analysts Nos. 1, 4 and 8 determined antimony by FAAS after extraction in 4-methylpentan-2-one/TOPO from an iodide solution[2]. Nos. 2, 10 and 11 used ETAAS directly on acid solutions[1].

Analyst No. 4 also determined antimony by ETAAS directly on an acid solution and found 47.0µg/g.

ARSENIC

Analysts Nos. 1 and 15 extracted arsenic as iodide[3] and chloride respectively into chloroform and completed photometrically as molybdenum blue. Nos. 2 and 10 used ETAAS directly on an acid solution[1]. 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 40.0µg/g. No. 2 also used a photometric method involving absorption of arsine in silver diethyldithiocarbamate/ephedrine in chloroform and found 54.8µg/g.

CADMIUM

Analysts Nos. 1, 4, 5 and 12 determined cadmium by ETAAS directly on acid solutions[1]. No. 2 used ETAAS 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 Flame Atomic Emission Spectrometry (FAES). No. 4 used a photometric method with brilliant green. No. 7 used ETAAS directly on an acid solution. Analyst No. 15 determined gallium by ETAAS directly on an acid solution[1].

TIN

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

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 ETAAS 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, Kriege 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

INTENDED USE

SS-CRM 346A 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

SS-CRM 346A will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surface).

TRACEABILITY

The characterisation of SS-CRM 346A 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 SS-CRM 346A 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 has been assessed on representative samples taken from the batch 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

In the case of SS-CRM 346A 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.P. MEERES, Managing Director

Preliminary Edition .... October 1982
Main Edition (revised with values for thallium, arsenic, gallium, and calcium and C(95%), s_M and s_W values for each certified element) .... June 2003
Main Edition (revised with editorial changes) .... October 2019