MAIN EDITION

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 178/2

TIN-BASE WHITE METAL

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

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	Pb	Sn	Sb	Cu	As	Bi	Cd	Fe	Ni	Zn	*Al*	*Ag*
1	3.18	82.2	9.40	4.57	0.14	0.10	0.14	0.024	0.17	0.038	*0.002*	...
2	3.12	82.3	9.51	4.58	0.12	0.10	0.14	0.022	0.17	0.039	*0.003*	...
3	3.16	81.9	9.51	4.55	0.17	0.09	0.13	0.023	0.17	0.043	*0.003*	...
4	3.24	82.3	9.36	4.63	0.19	0.13	0.14	0.024	0.16	0.039	...	...
5	3.17	82.4	9.44	4.56	0.16	0.12	0.13	0.025	0.18	0.040	*0.008*	...
6	3.19	82.2	9.53	4.56	0.12	...	...	...	0.17	...	...	...
7	3.18	82.1	9.43	4.56	0.14	0.11	0.14	0.023	0.18	0.041	*0.003*	*0.021*
8	3.16	82.0	9.43	4.64	0.15	0.10	0.14	0.024	0.19	0.039	*0.01*	...
M_M	**3.18**	**82.2**	**9.45**	**4.58**	**0.15**	**0.11**	**0.14**	**0.024**	**0.17**	**0.040**	...	...
s_M	0.04	0.2	0.06	0.04	0.03	0.02	0.01	0.001	0.01	0.002	...	...

The above figures are those which each 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 %

	Pb	Sn	Sb	Cu	As	Bi	Cd	Fe	Ni	Zn
Cv	**3.18**	**82.2**	**9.45**	**4.58**	**0.15**	**0.11**	**0.14**	**0.024**	**0.17**	**0.040**
C(95%)	0.03	0.2	0.06	0.03	0.03	0.02	0.01	0.001	0.01	0.002

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 178/2 TIN-BASE WHITE METAL

NOTES ON METHODS USED

LEAD

Analysts Nos. 1, 3, 4, 5, 7 and 8 determined lead electrolytically as the dioxide. Nos. 1, 3, 4 and 5 used the Standard method BS 3338: Part 15: 1965. No. 2 determined lead gravimetrically as lead molybdate after removal of tin, antimony and arsenic as the volatile bromides and separation as lead sulphate. Analyst No. 6 removed tin, antimony and arsenic as the volatile bromides, separated lead as the sulphate and titrated with EDTA.

Analysts Nos. 4, 7 and 8 also determined lead by atomic absorption spectroscopy and found 3.24%, 3.14% and 3.17% respectively.

TIN

All Analysts determined tin by titration. Nos. 1, 2, 3, 4, 6 and 8 dissolved the sample in bromine-hydrochloric acid, separated antimony by double precipitation with iron powder, reduced with nickel and titrated with iodate/iodide solution (Nos. 1, 2, 3, 4 and 8) or iodine solution (No. 6). Nos. 1, 2, 3 and 4 used the Standard method for tin in white metal bearing alloys BS 3338: Part 16: 1965. No. 7 used a similar method but reduced with iron and titrated with iodine solution. No. 5 dissolved the sample in sulphuric acid, reduced with nickel and titrated with iodate/iodide according to the A.S.T.M. method Designation E57-60.

ANTIMONY

Analysts Nos. 1, 2, 3, 4 and 5 dissolved the sample in concentrated sulphuric acid, added hydrochloric acid, reduced with sodium sulphite, boiled to remove arsenic and titrated with potassium bromate solution. Nos. 1, 3 and 4 used the Standard method BS 3338: Part 13: 1965. Nos. 2 and 5 used the A.S.T.M. method Designation E57-60. No. 8 used a similar method but used a correction for arsenic. No. 7 dissolved the sample in concentrated sulphuric acid, added hydrochloric acid and titrated with potassium permanganate solution. Analyst No. 6 dissolved the sample in concentrated sulphuric acid, removed copper with hydrogen sulphide and titrated with methylene blue and titanous chloride solutions.

Analyst No. 8 also used a method involving dissolution in bromine-hydrochloric acid, reduction with sodium sulphite and titration with potassium bromate solution and found 9.51%.

COPPER

Analysts Nos. 1, 2, 3, 4, 5, 7 and 8 determined copper electrolytically after removal of tin, antimony and arsenic as the volatile bromides. No. 2 also removed the lead as the sulphate before electrolysis whereas Nos. 1, 3, 4, 5, 7 and 8 electrolysed from a nitric acid solution without prior removal of lead; Nos. 1, 3, 4 and 5 followed the procedure of the Standard method BS 3338: Part 15: 1965. Analyst No. 6 removed tin, antimony and arsenic as the volatile bromides, separated lead as the sulphate and titrated with sodium thiosulphate solution.

Analysts Nos. 4, 7 and 8 also determined copper by atomic absorption spectroscopy and found 4.63%, 4.54% and 4.66% respectively.

ARSENIC

Analyst No. 1 determined arsenic turbidimetrically as colloidal arsenic. Nos. 2, 3, 4, 5, 6 and 7 separated arsenic by distillation as the trichloride. Nos. 2 and 3 used the Standard photometric method BS 3338: Part 9: 1965 in which a portion of the distillate is converted to molybdenum blue. Nos. 4, 5 and 7 completed titrimetrically by titration of the distillate with potassium bromate, No. 4 according to the A.S.T.M. method Designation E46-66. No. 6 separated arsenic as sulphide and titrated with iodine solution. Analyst No. 8 used atomic absorption spectroscopy.

Analyst No. 1 also determined arsenic by atomic absorption spectroscopy and found 0.12%.

BISMUTH

Analysts Nos. 1, 4, 7 and 8 used atomic absorption spectroscopy. Nos. 2, 3 and 5 separated tin and antimony as the volatile bromides and determined bismuth photometrically with thiourea according to the Standard method BS 3338: Part 8: 1961.

CADMIUM

Analysts Nos. 1, 3, 4, 7 and 8 used atomic absorption spectroscopy. Nos. 2 and 5 determined cadmium polarographically after separation of tin, antimony and copper.

IRON

Analysts Nos. 1, 3, 4, 7 and 8 used atomic absorption spectroscopy. No. 2 separated tin, antimony and arsenic as the volatile bromides and completed photometrically with 1:10-phenanthroline. No. 5 separated interfering elements according to the A.S.T.M. method Designation E87-58 and completed photometrically as the thiocyanate. No. 7 also completed as the thiocyanate after first separating iron as the hydroxide.

NICKEL

Analysts Nos. 1, 2, 5 and 7 determined nickel photometrically with dimethylglyoxime, No. 5 according to the A.S.T.M. method Designation E87-58. No. 2 first separated nickel by extraction into chloroform. Nos. 3 and 8 used atomic absorption spectroscopy. Nos. 4 and 6 separated nickel by precipitation with dimethylglyoxime, No. 4 completing by titration with EDTA and No. 6 gravimetrically.

Analysts Nos. 4 and 7 also determined nickel by atomic absorption spectroscopy and found 0.16% and 0.18% respectively. No. 8 also used a dimethylglyoxime gravimetric method and found 0.20%.

ZINC

Analysts Nos. 1, 2, 3, 4, 5, 7 and 8 used atomic absorption spectroscopy, No. 2 first separated tin, antimony and arsenic; No. 5 separated tin, antimony, arsenic, lead and copper.

ALUMINIUM

Analysts Nos. 1, 2, 3, 5 and 8 determined aluminium photometrically, Nos. 1, 2 and 3 used the Standard method BS 3338: Part 18: 1966 which depends on the colour reaction with chrome azurol S. No. 5 used eriochrome cyanine after removal of interfering elements. No. 8 extracted the aluminium as the 8-hydroxyquinolate into chloroform. No. 7 used atomic absorption spectroscopy.

SILVER

Atomic absorption spectroscopy.

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded <180micron (85 mesh) for chemical analysis.

INTENDED USE & STABILITY

The sample 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.

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For BUREAU OF ANALYSED SAMPLES LTD

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