MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 374

PHOSPHOR BRONZE

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

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

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ANALYSTS representing MANUFACTURERS and USERS

3 BALDWIN, R. M., *LRIC,* Kent Alloys, Ltd., Rochester.

4 CRESSWELL, C., *ARIC,* British Insulated Callender's Cables Ltd., Prescot.

ANALYSTS representing MANUFACTURERS and USERS (cont.)

5 ELWELL, W. T., *PhD, FRIC,* Imperial Metal Industries Ltd., Birmingham.

6 MCCRACKAN, J.D., Anaconda American Brass Company, Waterbury, U.S.A.

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8 SALE, G., Thomas Bolton & Sons Ltd., Stoke-on-Trent.

ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	Cu	Sn	Zn	Pb	P	Ni	S	*Fe*	*Al*	*Sb*	*Si*	*Bi*
1	89.56	9.75	0.006	0.060	0.59	0.015	0.012	*<0.005*	*<0.005*	*0.01*	*<0.005*	*0.007*
2	…	9.77	0.006	0.061	0.58	0.014	0.012	*<0.002*	*<0.002*	...	...	...
3	89.41	9.83	0.006	0.059	0.61	0.014	…	*0.004*	...	...	...	...
4	89.46	9.80	0.005	0.068	0.60	0.015	0.011	...	...	...	*<0.005*	...
5	89.51	9.88	0.006	0.060	0.59	0.015	0.014	*0.002*	*<0.002*	...	*<0.005*	...
6	89.54	9.81	0.006	0.066	0.57	0.013	0.014	*<0.001*	*<0.001*	...	*<0.001*	*0.007*
7	89.5	9.68	0.007	0.068	0.59	…	0.012	*0.005*	...	...	...	...
8	89.51	9.89	0.007	0.070	0.60	0.015	0.011	*<0.003*	*<0.003*	...	*<0.005*	...
M_M	**89.5**	**9.80**	**0.006**	**0.064**	**0.59**	**0.014**	**0.012**	*<0.005*	*<0.005*	*0.01*	*<0.005*	*0.007*
s_M	0.1	0.07	0.001	0.005	0.02	0.001	0.002	...	...	...	...	...

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 intralaboratory means. s_M: standard deviation of the intralaboratory means.

CERTIFIED VALUES (Cv)

mass content in %

	Cu	Sn	Zn	Pb	P	Ni	S
Cv	**89.5**	**9.80**	**0.006**	**0.064**	**0.59**	**0.014**	**0.012**
C(95%)	0.1	0.06	0.001	0.004	0.02	0.001	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 374 PHOSPHOR BRONZE

NOTES ON METHODS USED

COPPER

All Analysts determined copper electrolytically Nos. 1, 3, 4 and 8 carried out a preliminary separation of the tin by evaporation with hydrobromic acid/bromine and electrolysed from a nitric solution. Nos. 5 and 7 separated the tin as metastannic acid; No. 5 volatilised the tin oxide with ammonium iodide and determined the residual copper by atomic-absorption spectroscopy. No. 6 electrolysed from a nitric/hydrofluoric acid solution without separation of tin.

Analyst No. 8 also electrolysed from a nitric/fluoroboric acid solution and found 89.50%.

TIN

All Analysts determined tin titrimetrically Nos. 1, 2 and 8 carried out a preliminary separation of the tin and reduced with metallic nickel according to the Standard method BS 1748 Part 6 1960. Nos. 3, 5 and 7 reduced with hypophosphorous acid or sodium hypophosphite. Nos. 4 and 6 separated tin as metastannic acid and, after dissolving in hydrochloric acid, reduced with aluminium.

ZINC

Analyst No. 1 first removed tin by volatilisation as the bromide, then separated zinc by ion-exchange and determined it polarographically All the other Analysts determined zinc by atomic-absorption spectroscopy.

LEAD

Analysts No. 1 and 8 determined lead polarographically. No. 1 first removed tin by volatilisation as the bromide, then separated the lead from copper by precipitation with ammonia solution after the addition of a little iron as a carrier, the hydroxide precipitate was dissolved in hydrochloric acid and the iron reduced with ascorbic acid before polarographing. No. 8 eliminated interference from tin by dissolving in nitric acid and separated the copper as oxalate. The other Analysts determined lead by atomic-absorption spectroscopy.

PHOSPHORUS

Analysts Nos. 1, 2 and 3 determined phosphorus titrimetrically after precipitation of ammonium phosphomolybdate. No. 1 used the Analoid method No. 28. Nos. 4, 5, 6, 7 and 8 determined phosphorus photometrically as phosphovanadomolybdic acid, Nos. 4 and 8 used the Standard method BS 1748 Part 8 1960, No. 6 used the ASTM method Designation E 62.

NICKEL

Analysts Nos. 1, 2, 3, 4 and 8 determined nickel photometrically with dimethylglyoxime after separation of copper by electrolysis. No. 1 separated nickel from the copper-free electrolyte by extraction of the dimethylglyoxime complex into chloroform, then extracted the complex back into the aqueous phase and determined nickel in the usual way. Nos. 2, 3, 4 and 8 carried out the nickel determination directly on the copper-free electrolyte; Nos. 4 and 8 used the Standard method BS 1748 part 5 1961 Nos. 5 and 6 determined nickel by atomic-absorption spectroscopy.

SULPHUR

Analyst No. 1 determined sulphur gravimetrically as $BaSO_4$ after removal of tin by evaporation with hydrobromic acid/bromine followed by chromatographic separation of sulphur, as sulphuric acid, on an alumina column (Nydahl, Anal. Chem., 1954, **26**, 580). No. 2 used an evolution method in which the sulphur was evolved as hydrogen sulphide from a hydrobromic acid solution and determined photometrically as methylene blue. Nos. 4, 5 and 6 determined sulphur by combustion in oxygen. Nos. 4 and 6 completed titrimetrically. No. 5 completed photometrically with rosaniline and formaldehyde (Barabas and Kaminski, Anal. Chem., 1963, **35**, 1702). No. 7 distilled the sulphur as hydrogen sulphide from hydriodic acid/hypophosphorus acid and titrated with iodine/sodium thiosulphate. No. 8 determined sulphur by combustion in hydrogen, the hydrogen sulphide formed being absorbed in cadmium acetate solution and determined iodimetrically.

Analyst No. 1 also used a combustion method and found 0.013%.

IRON

Analysts Nos. 1, 3, 5, 6 and 8 determined iron photometrically after separation of copper. Nos. 1, 3 and 8 used 1, 10-phenanthroline, Nos. 5 and 6 used thioglycollic acid. Nos. 2 and 7 determined iron by atomic-absorption spectroscopy.

ALUMINIUM

Analysts Nos. 1, 2, 3 and 8 determined aluminium photometrically. Nos. 1 and 5 used eriochrome cyanine, No. 2 used chrome-azurol S and No. 8 used aluminon according to the Standard Method BS 1748 Part 4: 1961. No. 6 determined aluminium spectrographically after melting a chip sample under argon.

ANTIMONY

Copper was separated as cuprous iodide and antimony determined photometrically as the iodoantimonite complex.

SILICON

Analysts No. 1, 4 and 8 determined silicon photometrically as molybdenum-blue according to the Standard method BS 1748: Part 7: 1960. No. 5 used a molybdenum-blue method after distillation of the silicon as fluorosilicic acid. No. 6 determined silicon spectrographically after melting a chip sample under argon.

BISMUTH

Both Analysts determined bismuth photometrically as the iodide. No. 1 first separated copper as cuprous iodide and determined bismuth in the filtrate. No. 6 separated bismuth by chloroform extraction of the diethyldithiocarbamate complex from EDTA-cyanide solution.

DESCRIPTION OF SAMPLE

Bottles of 100g chips graded 1700-250 micron (10-60 mesh).

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.

TRACEABILITY

The traceability of BCS-CRM 374 to the SI is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

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For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
Managing Director

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