MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 364
LEADED BRONZE

BS 1400: 1969, LB2

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

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	Cu	Sn	Zn	Pb	P	Ni	Sb	*Fe*	*Al*	*As*	*Si*	*Bi*	*S*
1	80.60	9.35	0.12	9.25	0.055	0.30	0.18	*<0.005*	*0.001*	*0.065*	*<0.002*	*0.009*	*0.062*
2	80.70	9.35	0.13	9.10	0.055	0.28	0.18	*0.005*	...	*0.08*	...	...	*0.052*
3	80.65	9.40	0.12	9.20	0.060	0.27	0.19	*<0.002*	*<0.002*	*0.063*	...	...	*0.070*
4	80.70	9.40	0.12	9.10	0.055	0.30	0.16	*<0.002*	...	*0.065*	...	*0.01*	*0.055*
5	80.75	9.35	0.12	9.35	0.056	0.29	...	*0.003*	*<0.001*	...	*0.004*	...	...
6	80.55	9.25	0.14	9.25	0.056	0.28	0.18	*0.005*	*0.002*	...	...	...	...
7	80.65	9.30	0.12	9.35	0.057	0.27	0.17	*0.002*	*<0.002*	...	*<0.002*	...	...
8	80.65	9.50	0.14	9.30	0.058	0.28	0.17	*<0.002*	*<0.001*	...	*<0.001*	*0.007*	...
M_M	**80.7**	**9.36**	**0.13**	**9.24**	**0.057**	**0.28**	**0.18**	*<0.005*	*<0.002*	*0.07*	*<0.005*	*<0.01*	*0.06*
s_M	0.1	0.08	0.01	0.10	0.002	0.02	0.01	...	...	...	...	...	...

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	Sb
Cv	**80.7**	**9.36**	**0.13**	**9.24**	**0.057**	**0.28**	**0.18**
C(95%)	0.1	0.07	0.01	0.09	0.002	0.01	0.01

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 364 LEADED BRONZE

NOTES ON METHODS USED

COPPER

All Analysts determined copper electrolytically. No. 1 carried out a preliminary separation of tin by evaporation with hydrobromic acid/bromine and then separated lead as sulphate before determining the copper. Nos. 2 and 3 electrolysed from a nitric acid solution after filtering off the metastannic acid; any copper occluded in the metastannic acid was recovered. Nos. 4 and 8 electrolysed from a nitric/fluoroboric acid mixture. Nos. 5 and 7 electrolysed from a nitric acid solution after separation of tin by evaporation with hydrobromic acid/bromine according to one or other of the Standard methods BS 1748: Part 1 1961 or BS 1748: Part 11: 1964.

Analyst No. 2 used two alternative methods; (a) electrolysis from nitric/fluoroboric acid which gave a result of 80.70%. (b) titrimetric determination with sodium thiosulphate solution which gave a result of 80.80%. No. 8 also electrolysed from a nitric acid solution after filtration of metastannic acid and found 80.65%.

TIN

Analysts Nos. 1, 2 and 8 used the Standard titrimetric method BS 1748: Part 6: 1960 in which the tin is separated from copper by double precipitation with ammonia before reducing with a nickel coil and titrating with iodide/iodate solution. No. 3 used a similar method. No. 4 used both the British Standard method and a titrimetric method involving reduction with hypophosphite. Nos. 5 and 6 dissolved the sample in hydrochloric acid/hydrogen peroxide and reduced with sodium hypophosphite before titrating with iodide/iodate. No. 7 carried out a preliminary separation by precipitation with ammonia according to the British Standard method and then reduced with hypophosphite and titrated with iodide/iodate.

Analyst No. 3 also determined tin photometrically with quercetin in ethanol after addition of thiourea and potassium iodide; the result by this method was 9.40%. No. 5 also used the British Standard method and found 9.35%

ZINC

Analysts Nos. 1, 5 and 8 determined zinc polarographically after separation of interfering elements. Nos. 1 and 5 first removed tin by evaporation with hydrobromic acid/bromine and then separated zinc by ion-exchange according to the Standard method BS 1748: Part 9: 1963. Nos. 2, 4 and 7 determined zinc by atomic-absorption spectroscopy*. No. 3 separated the bulk of the interfering elements by precipitation with thiosulphate and determined zinc photometrically with dithizone. No. 6 determined zinc gravimetrically as oxinate.

. Analyst No. 2 also determined zinc by two gravimetric methods; (a) copper, tin and lead were removed as sulphides from an acid solution, the zinc precipitated as sulphide from a buffered formate solution and ignited to zinc oxide, giving a result of 0.11%. (b) the zinc was separated as sulphide but was dissolved in brominated hydrochloric acid and re-precipitated as zinc mercuric thiocyanate, giving a result of 0.12%. No. 5 also used atomic-absorption spectroscopy and found 0.13%. Nos. 5 and 6 determined zinc titrimetrically with EDTA and found 0.12% and 0.14% respectively.

LEAD

Analyst No. 1 carried out a preliminary separation of tin by evaporation with hydrobromic acid/bromine and then determined lead gravimetrically by separation as the sulphate and conversion to the molybdate*. No. 2 determined lead by atomic-absorption spectroscopy. Nos. 3, 6, 7 and 8 determined lead gravimetrically after anodic deposition as PbO_2. Nos. 3 and 6 weighed the deposited PbO_2 whereas Nos. 7 and 8 dissolved the deposit and converted to lead molybdate according to the Standard method BS 1748: Part 2: 1961. No. 4 used two methods (a) deposition as PbO_2 followed by solution of the deposit and titration with EDTA (b) gravimetric determination as $PbSO_4$. No. 5 separated the lead by deposition as PbO_2, dissolved the deposit and determined the lead titrimetrically with EDTA according to the Standard method BS 1748: Part 11: 1964.

Analyst No. 2 also separated lead by anodic deposition, dissolved the deposit and determined the lead iodimetrically; the result was 9.05%. No. 3 used two alternative methods (a) gravimetric determination as the sulphate which gave a result of 9.05%, (b) solvent extraction of the lead as the diethyldithiocarbamate followed by EDTA titration which gave a result of 9.10%.

PHOSPHORUS

Analysts Nos. 1, 3, 4, 5, 7 and 8 determined phosphorus photometrically as phosphovanadomolybdic acid. Nos. 1, 4, 5 and 8 used the Standard method BS 1748: Part 8: 1960. No. 7 extracted the coloured complex into *iso*-amyl alcohol before measuring the absorbance*. Nos. 2 and 6 determined phosphorus titrimetrically via the phosphomolybdate after removal of arsenic by evaporation with hydrobromic acid.

Analyst No. 1 also determined phosphorus titrimetrically after removal of arsenic with hydrobromic acid and found 0.055%.

NICKEL

All Analysts determined nickel by means of dimethylglyoxime in the electrolyte remaining after separation of copper. Nos. 1, 4, 5 and 7 used photometric methods, Nos. 2, 3, 6 and 8 used gravimetric methods.

Analyst No. 3 also determined nickel photometrically with dimethylglyoxime after separation of copper as cuprous iodide and found 0.28%. Nos. 2, 6 and 7 also determined nickel by atomic-absorption spectroscopy and found 0.28%, 0.28% and 0.27% respectively.

ANTIMONY

All Analysts except No. 6 determined antimony photometrically. Nos, 1, 3, 4 and 8 used methods based on the iodoantimonite complex. Nos. 1, 4 and 8 first separated copper by precipitation as cuprous iodide whereas No. 3 separated the antimony. by co-precipitation with MnO_2.: No. 2 separated the tin and antimony oxides by filtration from a nitric acid solution, dissolved in sulphuric/hydrochloric acid, extracted antimony with isopropyl ether and determined it photometrically with Rhodamine B. No. 7 used a similar method but without any preliminary separation*. No. 6 determined antimony titrimetrically with potassium permanganate solution.

IRON

All Analysts except Nos. 7 and 8 determined iron photometrically. Nos. 1, 4, 5 and 6 used 1: 10 - phenanthroline methods on the solution remaining after electrolytic separation of copper; No. 5 followed the procedure of the Standard method BS 1748: Part 3: 1961. No. 2 used a thiocyanate method. No. 3 used sulphosalicyclic acid in a solution of controlled acidity. No. 7 used atomic-absorption spectroscopy. No. 8 determined iron spectrographically by total volatilisation of a weighed sample in a DC arc.

ALUMINIUM

All Analysts except No. 8 determined aluminium photometrically. Nos. 1, 3, 6 and 7 used the reaction with eriochrome cyanine. Nos. 1 and 7 carried out preliminary separations by evaporation with hydrobromic acid/bromine and mercury-cathode electrolysis. No. 3 carried out a preliminary separation with sodium thiosulphate and used nitrated eriochrome cyanine for the colour reaction. No. 6 used a direct method without any preliminary separations. No. 8 determined aluminium spectrographically by total volatilisation of a weighed sample in a DC arc.

ARSENIC

Analysts Nos. 1 and 2 determined arsenic turbidimetrically after reduction to elemental arsenic with hypophosphite. No. 3 separated the arsenic by precipitation with hypophosphite, dissolved the precipitate in iodine solution and titrated excess iodine with standard arsenite solution. No. 4 determined arsenic photometrically as molybdenum-blue.

SILICON

Analysts Nos. 1, 5 and 7 determined silicon photometrically as molybdenum-blue according to the Standard method BS 1748 Part 7: 1960. No. 8 determined silicon spectrographically by total volatilisation of a weighed sample in a DC arc.

BISMUTH

Colorimetric determination as bismuth iodide.

SULPHUR

All Analysts used combustion methods.

*Elwell and Scholes, Analysis of Copper and its Alloys, Pergamon Press, 1967.

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 364 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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