MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 177/2

LEAD-BASE WHITE METAL

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

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

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6 MANDER, P. M., Fry's Metals Ltd., London.
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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	Pb	Sn	Sb	Cu	As	Bi	Ni
1	84.7	4.99	10.22	0.12	...	0.024	0.006
2	84.4	5.07	10.02	0.116	0.04	0.030	0.008
3	84.4	5.12	10.15	0.12	0.04	0.03	0.007
4	84.5	5.10	10.08	0.109	0.06	0.024	0.006
5	84.5	5.02	10.01	0.120	0.05	0.032	0.007
6	84.6	5.16	10.00	0.125	0.05	0.029	0.007
7	84.5	5.06	10.06	0.12	0.05	0.029	0.008
8	...	...	...	...	0.05	...	...
M_M	**84.5**	**5.07**	**10.1**	**0.12**	**0.05**	**0.028**	**0.007**
s_M	0.2	0.06	0.1	0.01	0.01	0.004	0.001

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

The following additional information was supplied by Analyst No. 4:- Ag 0.008%.

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	Ni
Cv	**84.5**	**5.07**	**10.1**	**0.12**	**0.05**	**0.028**	**0.007**
C(95%)	0.1	0.06	0.1	0.01	0.01	0.003	0.001

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.

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NOTES ON METHODS USED

LEAD

Analysts Nos. 1 and 2 removed tin, antimony and arsenic as the volatile bromides and determined lead by titration with EDTA solution. Nos. 3 and 4 carried out a preliminary separation of lead as sulphate and completed by titration with EDTA solution. Analysts Nos. 5 and 7 determined lead gravimetrically, No. 5 as lead molybdate and No. 7 as lead sulphate. No. 6 removed tin, antimony and arsenic as the volatile bromides and determined lead electrolytically as the dioxide.

Analyst No. 4 also determined lead by direct titration with ammonium molybdate solution and found 84.4%.

TIN

All Analysts determined tin by titration with iodine or iodate solution. No. 2 dissolved the sample in a mixture of hydrogen peroxide and acetic acid, removed antimony by boiling with iron powder, reduced with nickel and titrated with iodate. Nos. 1, 3, 4 and 7 dissolved the sample in brominated hydrochloric acid, reduced with nickel and titrated with iodate. Nos. 1 and 4 followed the procedure of the Standard Method BS 3338: Part 16: 1965. No. 6 dissolved the sample in sulphuric acid, reduced with iron and titrated with iodine solution.

ANTIMONY

All Analysts determined antimony by titration. Nos. 1, 2, 3, 5 and 6 dissolved the sample in sulphuric acid, added hydrochloric acid, reduced with sodium sulphite, boiled to remove arsenic and titrated with potassium bromate solution (Nos. 1, 2, 3 and 5) or potassium permanganate solution (No. 6). No. 2 followed the procedure of the ASTM method Designation E57-60. Nos. 4 and 7 dissolved the sample in a mixture of hydrochloric acid and bromine, reduced with sodium sulphite and titrated with potassium bromate solution.

COPPER

Analysts Nos. 1, 2, 3, 4 and 7 used atomic absorption spectroscopy. No. 5 determined copper titrimetrically with thiosulphate. No. 6 dissolved the sample in bromine-hydrobromic acid and completed photometrically as the bromide.

No. 7 also determined copper photometrically as the bromide and found 0.12%.

ARSENIC

Analysts Nos. 2, 5 and 6 separated arsenic by distillation as the trichloride. No. 2 used the Standard photometric method BS 3908: Part 2: 1967 in which a portion of the distillate is converted to molybdenum blue. Nos. 5 and 6 completed by titration of the distillate with potassium bromate. No. 3 separated arsenic by precipitation with sodium hypophosphite, dissolved in excess standard iodine solution and back titrated with standard sodium arsenite. Nos. 4, 7 and 8 used atomic absorption spectroscopy.

Analyst No. 5 also used a photometric molybdenum blue method after separation by distillation and found 0.05%.

BISMUTH

Analysts Nos. 1, 3, 4, 6 and 7 determined bismuth by atomic absorption spectroscopy. Analysts Nos. 2 and 5 determined bismuth photometrically as the bromide according to the Standard Method BS 3908: Part 3: 1967.

NICKEL

Analysts Nos. 2 and 5 determined nickel photometrically with dimethylglyoxime. Nos. 1, 3, 4, 6 and 7 used atomic absorption spectroscopy.

No. 6 also determined nickel by a dimethylglyoxime photometric method and found 0.007%.

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded <104micron (140 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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Managing Director

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