MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 242/2 (ECRM 555-1)

FERRO-TUNGSTEN

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.	C	Si	Al	Sn	W	*S*
1	0.025	1.77	0.14	0.030	79.7	*0.019*
2	0.026	1.72	0.13	0.035	79.9	...
3	0.021	1.66	0.14	0.030	79.9	...
4	0.027	1.67	0.15	0.038	79.9	...
5	0.026	1.77	0.16	0.037	79.8	...
6	...	1.84	0.14	0.033	80.0	...
7	0.025	1.81	...	0.037	79.9	*0.016*
M_M	**0.025**	**1.75**	**0.14**	**0.034**	**79.9**	...
s_M	0.003	0.07	0.02	0.004	0.1	...

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.

During the examination of this sample by laboratories in the EEC the following additional information was obtained:

Phophorus - mean value of 0.024% from 4 laboratories. Iron - mean value of 15.2% from 6 laboratories

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Al	Sn	W
Cv	**0.025**	**1.75**	**0.14**	**0.034**	**79.9**
C(95%)	0.003	0.07	0.02	0.004	0.1

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.

DESCRIPTION OF SAMPLE

British Chemical Standard – bottles of 100g of finely divided material graded 150µm (100 mesh) for chemical analysis.

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NOTES ON METHODS USED

CARBON

All Analysts determined carbon by combustion in oxygen using either red lead or a mixture of pure iron and pure tin as flux. Nos. 1 and 4 completed by non-aqueous titration (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399). No. 2 used the conventional low pressure method (Cook and Speight, Analyst, 1956, **81**, 144) whereas No. 5 used a modification of this method (Dunnill and Kent, Metallurgia, 1970, **81**, 125). Nos. 3 and 7 completed by infrared absorption.

Analyst No. 2 also used an infrared absorption method and found 0.026%.

SILICON

Analysts Nos. 1, 5 and 6 determined silicon by titration after precipitation as potassium silicofluoride; Nos. 1 and 6 followed the procedure of the ASTM Method Designation E31-63. Nos. 2, 4 and 7 determined silicon gravimetrically. No. 2 fumed with a mixture of sulphuric, phosphoric and perchloric acids and took up the residue in tartaric acid solution. No. 4 fumed with sulphuric acid and took up the residue in oxalic acid solution; the solution was made ammoniacal and then acidified again before filtering off the silica. No. 7 fumed with a mixture of phosphoric acid and perchloric acid. No. 3 determined silicon by atomic-absorption spectroscopy.

Analysts Nos. 3 and 5 also determined silicon gravimetrically and found 1.63% and 1.77% respectively.

ALUMINIUM

Analysts Nos. 1 and 6 determined aluminium by titration via the oxinate; both Analysts first separated tungsten as tungstic oxide and then carried out a sodium hydroxide separation to remove iron. Nos. 2 and 5 determined aluminium photometrically with eriochrome-cyanine after removal of tungsten. No. 3 mixed the sample with graphite containing cobalt as internal standard and analysed by AC arc spectroscopy. No. 4 determined aluminium by atomic-absorption spectroscopy.

Analyst No. 2 also determined aluminium by titration via the oxinate and found 0.15%.

TIN

All Analysts except No. 3 determined tin by titration. Nos. 1, 2, 4, 5 and 7 carried out a preliminary separation of the tin as sulphide according to the British Standard Tin Method 2*. No. 3 mixed the sample with graphite containing cobalt as internal standard and analysed by AC arc spectroscopy.

TUNGSTEN

All Analysts decomposed the sample with hydrofluoric/nitric acid mixture and determined tungsten gravimetrically as the oxide. In most cases cinchonine was added to aid precipitation and in all cases a correction was applied for any iron in the final precipitate by fusing with sodium carbonate, leaching with water and weighing the insoluble residue. After filtering off the tungstic oxide precipitate, No. 2 determined the small amount of soluble tungsten in the filtrate by the photometric toluene-3:4-dithiol method.

SULPHUR

Both Analysts detemined sulphur by combustion.

*Methods for Sampling and Analysis of Iron, Steel and Other Ferrous Metals, B.S. Handbook No. 19, first published in 1970 by the BSI, 389 Chiswick High Road, London. W4 4AL.

INTENDED USE & STABILITY

The finely divided sample, BCS-CRM 242/2 (ECRM 555-1), 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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