MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

85X SSCH (batch A)

Certified Reference Material Information

Type:	LEAD / ANTIMONY / TIN (CAST)
Form and Size:	Disc 40mm Diameter x 15mm Thickness
Produced by:	MBH Analytical Limited
Certified and supplied by:	MBH Analytical Limited

Certified Analysis

Percentage element by weight

Element	Sb	Sn	Bi	Se	As	Cu	Ni
Value [1]	**5.52**	**2.64**	**0.0441**	**(0.015)**	**0.208**	**0.177**	**0.010**
Uncertainty [2]	0.06	0.04	0.0017	-	0.006	0.002	0.001

Element	Cd	Te	Ag	Fe	Zn	S
Value [1]	**0.0040**	**0.0070**	**0.0134**	**(0.002)**	**0.0007**	**0.0035**
Uncertainty [2]	0.0003	0.0008	0.007	-	0.0001	0.0003

Note: values given in parentheses are not certified - they are provided for information only.

Definitions

[1] The certified values are the present best estimates of the true content for each element. Each value is a panel consensus, based on the averaged results of an interlaboratory testing programme, detailed on page 3.

[2] The uncertainty values are generated from the 95% confidence interval derived from the wet analysis results, in combination with a statistical assessment of the homogeneity data, as described on page 2.

Certified by:

on 17th June 2005

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial-grade lead, tin and antimony; the other major alloying ingredients and trace elements were added as single elements or as binary alloys. The melt was cast by sequential transfer of aliquots into iron moulds. 2mm has been removed from the working face to minimise any surface effects.

Sampling

Samples for chemical analysis were taken from various positions throughout the casting process. Approximately 10% of all discs were selected for non-destructive homogeneity testing.

Homogeneity

The discs were checked for sample and batch uniformity using an optical emission spectrometer.

Using the meaned data from each surface, standard deviation values were derived for each element as an indicator of any non-homogeneity (as determined for the specific sample size taken by the spectrometer).

Chemical Analysis

Analysis was carried out on millings taken from samples representative of the product. It was performed by a panel of laboratories mostly operating within the terms of EN ISO/IEC 17025 - 2000, using documented standard reference methods and validated by appropriate reference materials.

The individual values listed overpage are the average of each analyst's results.

Estimation of Uncertainties

Each element certified has been analysed by several laboratories, and 95% half-width confidence intervals ($C_{(95\%)}$) for the resultant mean values have been derived by the method shown on page 3.

As a separate exercise, the degree of non-homogeneity of the batch for each element has been quantified by a programme of non-destructive application testing, discussed above.

The final certified uncertainty for each element has been derived by combining these two factors, using the square-root of the summed squares.

Traceability

Most of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to a national authority. It is part of the requirement for this accreditation that analytical work should be performed with due traceability, via an unbroken chain of comparisons, each with stated uncertainty, to primary standards such as the mole, or to nationally- or internationally-recognised primary reference materials.

Usage

Intended use:	With optical emission and X-ray fluorescence spectrometers.
Recommended method of use:	Lead and its alloys are generally prepared by machining on a lathe. However, users are recommended to follow the calibration and sample preparation procedures specified by the relevant instrument manufacturer. Preparation should be the same for reference materials and the samples for test. A minimum of four consistent replicate analyses is recommended to provide the necessary sample size. Users are advised to check against possible bias between reference materials and production samples due to differences in metallurgical history, and be aware of possible inter-element effects.

Analytical Data

Percentage element by weight

Sample	Sb	Sn	Bi	Se	As	Cu	Ni
1	5.329	2.579	0.0391	0.0107	0.199	0.175	0.0095
2	5.465	2.58	0.0419	0.0109	0.199	0.175	0.0097
3	5.468	2.612	0.0424	0.0112	0.200	0.176	0.0101
4	5.482	2.637	0.0427	0.0122	0.204	0.176	0.0103
5	5.50	2.657	0.0432	0.0126	0.206	0.177	0.0105
6	5.514	2.662	0.0449	0.0171	0.208	0.177	0.0106
7	5.547	2.666	0.0450	0.0171	0.210	0.177	0.0110
8	5.565	2.701	0.0452	0.0174	0.215	0.179	0.0115
9	5.570		0.0454	0.0178	0.217	0.180	
10	5.62		0.0458	0.0193	0.221	0.180	
11	5.64		0.0490				
Mean	**5.518**	**2.637**	**0.0441**	**0.0146**	**0.208**	**0.177**	**0.0104**
Std Dev	0.086	0.043	0.0026	0.0034	0.008	0.002	0.0007
$C_{(95\%)}$	0.058	0.036	0.0017	0.0024	0.006	0.002	0.0005

Sample	Cd	Te	Ag	Fe	Zn	S
1	0.0033	0.0056	0.0125	0.0008	0.0005	0.0031
2	0.0037	0.0058	0.0130	0.0010	0.0006	0.0032
3	0.0039	0.0064	0.0132	0.0010	0.0007	0.0033
4	0.0039	0.0065	0.0136	0.0016	0.00078	0.0034
5	0.0039	0.0067	0.0137	0.0019	0.0008	0.0035
6	0.0040	0.0073	0.0143	0.0024		0.0038
7	0.0040	0.0075		0.0031		0.0039
8	0.0043	0.0076		0.0032		
9	0.0047	0.0092				
Mean	**0.0040**	**0.0070**	**0.0134**	**0.0019**	**0.0007**	**0.0035**
Std Dev	0.0004	0.0011	0.0006	0.0009	0.0001	0.0003
$C_{(95\%)}$	0.0003	0.0008	0.007	0.0008	0.0001	0.0003

Note: $C_{(95\%)}$ is the 95% half-width confidence interval derived from the equation:

$$C_{(95\%)} = (t \times SD)/\sqrt{n}$$

where n is the number of available values, t is the Student's t value for n-1 degrees of freedom, and SD is the standard deviation of the test results.

Participating Laboratories

Bodycote Materials Testing Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office Ltd	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 492
Laboratory Testing Inc	Hatfield, PA, USA	A2LA accreditation 0117
Luo Yang Copper Co Ltd	Luo Yang, He Nan, China	CNAL accreditation 0173
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
RWTUV Laboratory	Brno, Czech Republic	CAI accreditation 1060
Coleshill Laboratories Ltd	Coleshill, England	
Spectroscopic Solutions Ltd	Western Cape, South Africa	
Advanced Analytical Centre, Cook University	Townsville, Qld, Australia	

Note: to achieve National Accreditation (eg UKAS, A2LA, NATA, CNAL, PCA, CAI), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

Analytical Methods Used

ELEMENT	RESULT No. & METHOD				
	ICP-AES	ICP-MS	FAAS	OTHER	
Antimony	1-4, 7-11	-	-	5, 6	volumetric (bromate)
Tin	1, 4-8	-	-	2, 3	volumetric (iodide)
Bismuth	1-5, 9-11	8	6, 7		
Selenium	1-3, 5, 7-10	4	6		
Arsenic	1-6, 9	7	8	10	photometric (turbidity)
Copper	1, 3-7, 9	2	8, 10		
Nickel	1-5, 7	-	6, 8		
Cadmium	1, 2, 4, 5, 8, 9	3	6	7	volumetric (iodate)
Tellurium	1-6, 8, 9	-	7		
Silver	1, 2, 6	5	3, 4		
Iron	1-6, 8	-	7		
Zinc	1-3	-	4, 5		
Sulfur	1, 2, 4	-	-	5	combustion (volumetric detection)
				3, 7	combustion (infra-red detection)
				6	OES

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, taking into account the requirements of ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The unidirectional solidification effects associated with semi-chill casting have led to the formation of inhomogeneous segregates in the rear portion of the disc. The above certification is therefore only applicable from the front face of the disc to a depth of 10mm. Material to the rear of the disc, to a depth of ~5mm, is not certified.

This material is liable to superficial corrosion, and there is some possibility of microstructural changes due to recrystallisation. However, it will otherwise remain stable provided adequate precautions are taken to protect it from cross-contamination, extremes of temperature and atmospheric moisture. All production records will be retained for a period of 20 years from the date of this certificate. This certification will therefore expire in June 2025, although we reserve the right to make changes as issue revisions, in the intervening period.

This sample is also available in the form of chippings.

The manufacture, analysis and certification of this product were supervised by C Eveleigh, PhD, Technical Director, MBH Analytical Ltd.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.