MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

85X A16 (batch A)

Certified Reference Material Information

Type:	LEAD / ANTIMONY (CAST)
Form and Size:	Disc 38-40mm diameter
Produced by:	Universal Scientific Laboratory Pty Ltd
Certified and supplied by:	MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Sn	Bi	Ag	Se	Sb	Cu
Value [1]	**0.0356**	**0.0165**	**0.0297**	**0.0218**	**1.57**	**0.0006**
Uncertainty [2]	0.0015	0.0007	0.0008	0.0007	0.02	0.0001

Element	Ni	S	As	Cd	Zn	Fe
Value [1]	**0.0006**	**(0.0003)**	**0.0503**	**0.00014**	**0.00011**	**(0.0001)**
Uncertainty [2]	0.0001	-	0.0010	0.00003	0.00002	-

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 2nd October 2014

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial ARA ingot to alloy designation A16, resistance melted under argon, with no additions. The melt was cast by sequential transfer of aliquots into individual iron moulds. Approximately 2mm has been removed from the working face of each disc, to minimise any surface effects.

Sampling

Samples for chemical analysis were taken from various positions throughout the casting process. At least 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 combined data for 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 - 2005, 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

Much of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to ISO 17025. It is an implicit 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 reference materials. In addition, some of the results derived as part of this testing programme have traceability to NIST standards, as part of the analytical calibration or process control.

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 five 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	Sn	Bi	Ag	Se	Sb	Cu
1	0.0332	0.0152	0.0283	0.0204	1.534	0.00035
2	0.0335	0.0153	0.0289	0.0204	1.535	0.00040
3	0.0338	0.0154	0.0291	0.0209	1.542	0.00047
4	0.0341	0.0156	0.0291	0.0211	1.557	0.00050
5	0.0348	0.0158	0.0292	0.0213	1.563	0.00050
6	0.0353	0.0158	0.0294	0.0214	1.569	0.00052
7	0.0354	0.0160	0.0299	0.0219	1.588	0.00060
8	0.0356	0.0161	0.0299	0.0223	1.589	0.00063
9	0.0357	0.0169	0.0299	0.0223	1.595	0.00070
10	0.0359	0.0171	0.0299	0.0225	1.599	0.00073
11	0.0364	0.0173	0.0305	0.0232	1.609	0.00074
12	0.0366	0.0178	0.0308	0.0238	1.618	0.00076
13	0.0372	0.0184	0.0313			0.00079
14	0.0377	0.0185				0.00080
15	0.0381					
Mean	**0.0356**	**0.0165**	**0.0297**	**0.0218**	**1.575**	**0.00061**
Std Dev	0.0015	0.0011	0.0008	0.0011	0.029	0.00015
$C_{(95\%)}$	0.0008	0.0007	0.0005	0.0007	0.018	0.00009

Sample	Ni	S	As	Cd	Zn	Fe
1	0.00040	0.00012	0.0491	0.00010	0.00006	0.00005
2	0.00045	0.00013	0.0497	0.00010	0.00007	0.00006
3	0.00046	0.00020	0.0499	0.00010	0.00009	0.00010
4	0.00050	0.00028	0.0501	0.00010	0.00010	0.00010
5	0.00057	0.00030	0.0501	0.00013	0.00010	0.00010
6	0.00059	0.00040	0.0501	0.00013	0.00010	0.00010
7	0.00060	0.00041	0.0503	0.00017	0.00012	
8	0.00063	0.00045	0.0504	0.00018	0.00013	
9	0.00065	0.00052	0.0507	0.00018	0.00013	
10	0.00066		0.0507	0.00019	0.00013	
11	0.00068		0.0509		0.00014	
12	0.00070		0.0520		0.00015	
13	0.00080				0.00015	
Mean	**0.00059**	**(0.0003)**	**0.0503**	**0.00014**	**0.00011**	**(0.0001)**
Std Dev	0.00011	-	0.0007	0.00004	0.00003	-
$C_{(95\%)}$	0.00007	-	0.0005	0.00003	0.00002	-

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

Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Birmingham Assay Office	Birmingham, England	UKAS accreditation 0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Bureau Veritas CPS Pvt Ltd	Chennai, India	NABL accreditation 0025
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
AIM Metals and Alloys LP	Montreal, Canada	
Raghavendra Spectrometallurgical Laboratory	Bangalore, India	
London & Scandinavian Met Co	Rotherham, England	
Laboratory Inppamet	Calama, Chile	
Coleshill Laboratories Ltd	Coleshill, England	
Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (UKAS, etc), 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
Tin	1, 3-5, 7, 8, 10-12, 14, 15	-	6, 9, 13	2	photometric (phenyl fluorone)
Bismuth	1, 3-6, 8-10, 14	-	2, 7, 12, 13	11	photometric (iodide)
Silver	1, 4-9	-	2, 3, 10-13		
Selenium	2-4, 6-11	-	1, 5, 12		
Antimony	1-3, 5-7, 12	-	4, 9, 10	8	photometric (crystal violet)
				11	volumetric (bromate)
Copper	1-5, 7, 8, 11, 13	9, 12	6, 10, 14		
Nickel	1, 2, 4, 6-9, 11, 13	3, 12	5, 10		
Sulfur	1, 7, 8	-	-	2-6, 9	combustion (infra-red detection)
Arsenic	1, 3, 5-9, 12	10	2, 11	4	photometric (molybdenum blue)
Cadmium	3, 4, 7, 9, 10	2, 5	1, 6, 8		
Zinc	3-6, 9-12	2, 7	1, 13		
Iron	2, 4-6	-	1, 3		

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 and ISO Guide 35-2006, taking into account the requirements of 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. The rear portion of the disc, to a depth of ~5mm, is not certified.

This material is liable to superficial corrosion. There is also a possibility for microstructural changes due to recrystallisation, and diffusion effects may lead to the concentration of some elements at the surface. For X-ray and other superficial sampling techniques, it is therefore recommended that the surface is refreshed immediately prior to use. In all other respects, this sample will remain stable indefinitely, 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. Technical support for this certification will therefore expire in October 2034, 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.