MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

85X 0494PB2 (batch C)

Certified Reference Material Information

Type:	LEAD ALLOY (CAST)
Form and Size:	Disc ~40mm diameter
Produced by:	Universal Scientific Laboratory Pty, Australia
Certified and supplied by:	MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Sb	Sn	S	Se	Fe
Value [1]	**1.928**	**0.124**	**0.0052**	**0.0272**	**(<0.001)**
Uncertainty [2]	0.017	0.003	0.0004	0.0010	-

Element	Te	Ag	As	Bi	Cu
Value [1]	**0.00382**	**0.0279**	**0.102**	**0.0387**	**0.0227**
Uncertainty [2]	0.00013	0.0010	0.002	0.0010	0.0006

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 3rd December 2017

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial lead, plus pure elements and master alloys, resistance melted under argon. 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 combined data for each surface, through-batch variation values were derived for each element as an indicator of any minor compositional variation (as determined for the specific sample size and other limitations of 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, 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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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	Sb	Sn	S	Se	Fe
1	1.898	0.1172	0.00450	0.0250	0.00010
2	1.907	0.1173	0.00480	0.0254	0.00010
3	1.909	0.1189	0.00500	0.0257	0.00012
4	1.911	0.1194	0.00510	0.0258	0.00016
5	1.921	0.1212	0.00511	0.0260	0.00025
6	1.928	0.1226	0.00518	0.0266	0.00029
7	1.930	0.1245	0.00520	0.0276	0.00050
8	1.946	0.1248	0.00578	0.0280	0.00053
9	1.963	0.1250	0.00608	0.0281	0.00055
10	1.963	0.1250		0.0282	0.00070
11		0.1250		0.0290	0.00083
12		0.1260		0.0307	0.00086
13		0.1271			
14		0.1292			
15		0.1306			
Mean	**1.928**	**0.1236**	**0.00520**	**0.0272**	**(0.00042)**
Std Dev	0.023	0.0041	0.00048	0.0017	-
$C_{(95\%)}$	0.017	0.0023	0.00037	0.0010	-

Sample	Te	Ag	As	Bi	Cu
1	0.00334	0.0252	0.0991	0.0350	0.0257
2	0.00350	0.0257	0.0992	0.0362	0.0264
3	0.00370	0.0260	0.0992	0.0365	0.0268
4	0.00371	0.0264	0.1001	0.0382	0.0270
5	0.00371	0.0264	0.1010	0.0382	0.0275
6	0.00375	0.0265	0.1013	0.0383	0.0275
7	0.00380	0.0265	0.1016	0.0384	0.0275
8	0.00380	0.0270	0.1024	0.0385	0.0278
9	0.00390	0.0272	0.1026	0.0385	0.0279
10	0.00390	0.0274	0.1038	0.0391	0.0279
11	0.00390	0.0287	0.1039	0.0395	0.0279
12	0.00400	0.0289	0.1040	0.0406	0.0281
13	0.00418	0.0292	0.1070	0.0406	0.0283
14	0.00419	0.0293	0.1070	0.0410	0.0291
15		0.0310		0.0419	0.0296
16		0.0311			0.0307
17		0.0314			
Mean	**0.00382**	**0.0279**	**0.1023**	**0.0387**	**0.0227**
Std Dev	0.00023	0.0020	0.0026	0.0019	0.0012
$C_{(95\%)}$	0.00013	0.0010	0.0015	0.0010	0.0006

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 Analytical Services	Sheffield, England	UKAS accreditation 0012
Anchorcert Analytical	Birmingham, England	UKAS accreditation 0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
TCR Engineering Services Ltd	Mumbai, India	
INCDMNR-IMNR	Pantelimon, Romania	
AMG Superalloys UK Ltd	Rotherham, England	
Coleshill Laboratories Ltd	Coleshill, England	
AIM Metals and Alloys LP	Montreal, Canada	
Laboratory Inppamet	Calama, Chile	
Analyticka Laborator Lithea sro	Brno, Czech Republic	
Johnson Controls Main Laboratory	Hannover, Germany	
Johnson Controls Plant Laboratory	Hannover, Germany	

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	
Antimony	3, 5, 7-10	-	1, 2, 4, 6		
Tin	3-8, 10, 11, 14, 15	1	2, 9, 13	12	photometric (phenyl fluorone)
Sulfur	1, 2, 5, 6, 9	-	-	3, 4, 7, 8	combustion (IR detection)
Selenium	1-4, 6-10, 12	5	11		
Iron	3-7, 10-13	8	1, 2, 9		
Tellurium	2-6, 8-13	14	1, 7		
Silver	4-8, 10, 11, 13-16	-	1-3, 9, 12, 17		
Arsenic	2-4, 6-13	1	14	5	photometric (molybdenum blue)
Bismuth	1, 4, 5, 7-13, 15	2	6, 14	3	photometric (iodide)
Copper	3, 6-16	2	1, 4, 5		

Notes

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

The unidirectional solidification effects associated with this method of 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. 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 December 2037, 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.