MBH ANALYTICAL LTD

HOLLAND HOUSE • QUEENS ROAD • BARNET • EN5 4DJ • ENGLAND • TEL: +44 (0)20 8441 2024 • FAX: +44 (0)20 8449 0810

email: info@mbh.co.uk web: www.mbh.co.uk

CERTIFICATE OF ANALYSIS

84X BA7 (batch B)

Certified Reference Material Information

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

Assigned Values

Percentage element by weight

Element	Sn	Sb	Bi	Cu	Cd	As
Value [1]	**0.594**	**0.0022**	**0.0140**	**0.0020**	**0.0004**	**(0.0004)**
Uncertainty [2]	0.004	0.0003	0.0005	0.0001	0.0001	-

Element	Ag	Zn	Al	Ni	Te	Ca
Value [1]	**0.0015**	**0.0024**	**0.0085**	**(0.0003)**	**(0.0002)**	**0.0391**
Uncertainty [2]	0.0001	0.0002	0.0007	-	-	0.0010

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 July 2013

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial Pb-Ca battery-alloy precursor and lead; the 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 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	Sb	Bi	Cu	Cd	As
1	0.585	0.0015	0.0124	0.0018	0.0003	0.0001
2	0.586	0.0015	0.0131	0.0018	0.0003	0.0002
3	0.586	0.0019	0.0131	0.0018	0.0003	0.0002
4	0.589	0.0020	0.0131	0.0018	0.0004	0.0004
5	0.590	0.0022	0.0136	0.0019	0.0004	0.0005
6	0.591	0.0022	0.0137	0.0019	0.0004	0.0005
7	0.591	0.0022	0.0138	0.0019	0.0004	0.0006
8	0.592	0.0023	0.0138	0.0020	0.0004	0.0008
9	0.595	0.0024	0.0141	0.0020	0.0005	
10	0.596	0.0025	0.0141	0.0023	0.0005	
11	0.598	0.0025	0.0147	0.0023	0.0005	
12	0.601	0.0026	0.0148	0.0023	0.0005	
13	0.603		0.0148	0.0024	0.0005	
14	0.605		0.0148		0.0005	
15	0.607		0.0155		0.0006	
Mean	**0.594**	**0.0022**	**0.0140**	**0.0020**	**0.00043**	**(0.0004)**
Std Dev	0.007	0.0004	0.0008	0.0002	0.00009	-
$C_{(95\%)}$	0.004	0.0002	0.0005	0.0001	0.00005	-

Sample	Ag	Zn	Al	Ni	Te	Ca
1	0.0011	0.0018	0.0067	0.0000	0.0000	0.0369
2	0.0012	0.0020	0.0070	0.0001	0.0000	0.0371
3	0.0012	0.0020	0.0076	0.0001	0.0001	0.0372
4	0.0012	0.0021	0.0076	0.0001	0.0002	0.0378
5	0.0012	0.0022	0.0081	0.0002	0.0002	0.0382
6	0.0013	0.0022	0.0081	0.0002	0.0003	0.0386
7	0.0015	0.0023	0.0083	0.0002	0.0003	0.0387
8	0.0015	0.0024	0.0083	0.0003	0.0003	0.0395
9	0.0015	0.0025	0.0090	0.0003	0.0004	0.0400
10	0.0016	0.0025	0.0091	0.0004	0.0004	0.0402
11	0.0016	0.0027	0.0092	0.0004		0.0403
12	0.0016	0.0028	0.0096	0.0005		0.0405
13	0.0018	0.0030	0.0099	0.0005		0.0409
14	0.0018	0.0030	0.0100			0.0409
15	0.0018					
Mean	**0.0015**	**0.0024**	**0.0085**	**(0.0003)**	**(0.0002)**	**0.0391**
Std Dev	0.0002	0.0004	0.0010	-	-	0.0015
$C_{(95\%)}$	0.0001	0.0002	0.0006	-	-	0.0008

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
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
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Sargam Metals 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	
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
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 (eg UKAS, NATA, etc), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

Analytical Methods Used

ELEMENT	RESULT No. & METHOD			
	ICP-AES	FAAS	OTHER	
Tin	1, 2, 7, 9-15	3, 5, 6, 8	4	photometric (phenylfluorone)
Antimony	1-3, 7-11	4, 5, 12	6	ICP-MS
Bismuth	1, 4, 6, 8-10, 12, 13, 15	2, 3, 5, 7, 14	11	photometric (iodide)
Copper	1-4, 7-11	5, 6, 12, 13		
Cadmium	2, 4-12	3, 13, 14	1, 15	ICP-MS
Arsenic	2-4, 6, 7	5, 8	1	ICP-MS
Silver	1-6, 10, 12, 14, 15	7-9, 11, 13		
Zinc	1-9, 11, 14	10, 12, 13		
Aluminium	1, 3, 6-8, 10, 12-14	4, 5, 11	2, 9	photometric (chrome azurol S)
Nickel	2-4, 7-11	1, 5, 12	6, 13	ICP-MS
Tellurium	1-4, 7-9	6	5, 10	ICP-MS
Calcium	1-3, 6, 7, 9-14	4, 5, 8		

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 applicable from the front face of the disc to a depth of 10mm. Material to the rear, 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 July 2033, 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.