MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

91X S40PR2 (batch D)

Certified Reference Material Information

Type:	LEAD-TIN SOLDER (CAST)
Form and Size:	Disc ~40mm diameter
Manufactured by:	MBH Analytical Ltd
Certified and Supplied by:	MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Sn	Sb	Bi	Cu	As
Value [1]	**40.68**	**0.596**	**0.154**	**0.085**	**0.010**
Uncertainty [2]	0.12	0.008	0.002	0.002	0.001

Element	Fe	Cd	Ag	Zn	Ni
Value [1]	**0.0096**	**0.0046**	**0.086**	**0.0275**	**0.0050**
Uncertainty [2]	0.0010	0.0002	0.004	0.0012	0.0005

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 30h September 2011

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial-purity lead and tin, pure elements, binaries and master alloys. The metal was cast from the bulk melt by sequential transfer of aliquots into individual iron chill moulds. At least 1mm has been machined from the working surface of each disc, to minimise 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 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 turnings taken from samples representative of the product. It was performed by participating laboratories mostly operating within the terms of EN ISO/IEC 17025 - 2005, using documented standard methods of analysis.

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 to method of use:	Solders are generally prepared by machining on a lathe. However, users are recommended 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	As
1	40.53	0.578	0.147	0.0818	0.0083
2	40.55	0.582	0.149	0.0825	0.0089
3	40.62	0.588	0.151	0.0839	0.0090
4	40.64	0.589	0.152	0.0846	0.0091
5	40.65	0.591	0.153	0.0849	0.0092
6	40.68	0.595	0.154	0.0849	0.0095
7	40.68	0.597	0.154	0.0856	0.0098
8	40.69	0.600	0.155	0.0865	0.0101
9	40.69	0.600	0.155	0.0872	0.0106
10	40.76	0.602	0.156	0.0887	0.0108
11	40.77	0.603	0.158	0.0892	0.0111
12	40.78	0.606	0.158		0.0111
13	40.84	0.608	0.159		0.0112
14		0.611			0.0121
Mean	**40.68**	**0.596**	**0.154**	**0.0854**	**0.0101**
Std Dev	0.09	0.010	0.004	0.0023	0.0011
$C_{(95\%)}$	0.05	0.006	0.002	0.0016	0.0006

Sample	Fe	Cd	Ag	Zn	Ni
1	0.0076	0.0042	0.0783	0.0258	0.0041
2	0.0082	0.0042	0.0790	0.0263	0.0044
3	0.0094	0.0044	0.0797	0.0264	0.0046
4	0.0094	0.0045	0.0801	0.0266	0.0046
5	0.0095	0.0045	0.0821	0.0270	0.0047
6	0.0096	0.0045	0.0874	0.0271	0.0048
7	0.0096	0.0046	0.0881	0.0272	0.0049
8	0.0097	0.0046	0.0885	0.0275	0.0050
9	0.0097	0.0046	0.0891	0.0279	0.0052
10	0.0098	0.0047	0.0919	0.0285	0.0052
11	0.0099	0.0048	0.0930	0.0290	0.0054
12	0.0111	0.0048	0.0956	0.0290	0.0055
13	0.0118	0.0050		0.0294	0.0060
14		0.0051			
Mean	**0.0096**	**0.0046**	**0.086**	**0.0275**	**0.0050**
Std Dev	0.0011	0.0003	0.006	0.0012	0.0005
$C_{(95\%)}$	0.0006	0.0002	0.004	0.0007	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

Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0114
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
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
AIM Metals and Alloys LP	Montreal, Canad	
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	Birmingham, England	

Note: to achieve the above-noted 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	2, 3, 5-9	13	1, 4, 11	volumetric (iodate)
			10	photometric (phenyl fluorone)
			12	gravimetric
Antimony	1-3, 5-8, 11, 13, 14	4, 10	9, 12	volumetric (bromate)
Bismuth	1, 2, 4, 5, 7-10	3, 6, 12, 13	11	photometric (iodide)
Copper	1, 2, 4-6, 8, 9	3, 7, 10, 11		
Arsenic	1, 3-8, 10, 12-14	2, 9, 11		
Iron	1, 4-9, 13	2, 3, 10-12		
Cadmium	2, 4, 6-9, 12-14	1, 3, 5, 10, 11		
Silver	1, 2, 4, 5, 7, 8, 12	2, 3, 6, 9-11		
Zinc	1-3, 5, 6, 9-11, 13	4, 7, 8, 12		
Nickel	2, 3, 5, 7, 9-11, 13	1, 4, 6, 8, 12		

Notes

This Certified Reference Material has been produced and certified, wherever possible, 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 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 maximum 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 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. This certification will therefore expire in September 2031, 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.