

# CERTIFICATE OF ANALYSIS

**91X S63PR0 (batch C)**

## Certified Reference Material Information

Type: LEAD-TIN SOLDER (CAST)  
Form and Size: Disc 40mm diameter x 15mm thick  
Manufactured by: MBH Analytical Ltd  
Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	Sn	Sb	Bi	Cu	As	Fe	Cd
Value <sup>1</sup>	60.02	0.0245	0.0101	0.0245	0.0170	0.0016	0.0124
Uncertainty <sup>2</sup>	0.12	0.0012	0.0006	0.0005	0.0014	0.0003	0.0006

Element	Ag	Zn	Ni	Au	In	Te	Hg
Value <sup>1</sup>	0.0201	(0.0004)	0.0039	(0.0004)	0.0098	(0.0005)	0.0082
Uncertainty <sup>2</sup>	0.0010	-	0.0003	-	0.0005	-	0.0004

Note: values in parentheses are not certified – they are provided for information only

## Definitions

- <sup>1</sup> 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.
- <sup>2</sup> 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:

MBH ANALYTICAL LIMITED \_\_\_\_\_ on 21<sup>st</sup> February 2019  
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. Approximately 1mm has been machined from the working surface of each disc, to minimise surface effects.

## **Sampling**

Milled samples for chemical analysis were taken from several positions within the batch. In addition, approximately 10% of all samples were selected for homogeneity checking.

## **Homogeneity**

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Multiple measurements were taken from each surface under test.

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 turnings taken from samples representative of the product. It was performed by participating laboratories mostly operating within the terms of EN ISO/IEC 17025, 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.

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: Solders 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	As	Fe	Cd
1	59.77	0.0222	0.0082	0.0222	0.0145	0.00090	0.0105
2	59.92	0.0225	0.0088	0.0236	0.0145	0.00091	0.0119
3	59.92	0.0227	0.0094	0.0236	0.0154	0.00104	0.0120
4	59.94	0.0236	0.0099	0.0242	0.0159	0.00123	0.0120
5	59.97	0.0238	0.0099	0.0242	0.0159	0.00150	0.0122
6	59.99	0.0243	0.0099	0.0243	0.0160	0.00150	0.0123
7	60.00	0.0248	0.0102	0.0244	0.0162	0.00150	0.0124
8	60.08	0.0254	0.0102	0.0244	0.0168	0.00160	0.0124
9	60.15	0.0254	0.0103	0.0244	0.0175	0.00170	0.0125
10	60.22	0.0255	0.0104	0.0248	0.0186	0.00184	0.0125
11	60.28	0.0266	0.0104	0.0252	0.0195	0.00200	0.0125
12		0.0274	0.0105	0.0253	0.0196	0.00210	0.0126
13			0.0108	0.0256	0.0203	0.00210	0.0129
14			0.0109	0.0256		0.00220	0.0131
15			0.0110	0.0257			0.0140
<b>Mean</b>	<b>60.02</b>	<b>0.0245</b>	<b>0.0101</b>	<b>0.0245</b>	<b>0.0170</b>	<b>0.00158</b>	<b>0.0124</b>
<b>Std Dev</b>	0.15	0.0016	0.0008	0.0009	0.0020	0.00044	0.0007
<b>C<sub>(95%)</sub></b>	0.10	0.0010	0.0004	0.0005	0.0012	0.00025	0.0004

Sample	Ag	Zn	Ni	Au	In	Te	Hg
1	0.0176	0.00013	0.0031	0.00014	0.0079	0.00023	0.0077
2	0.0189	0.00020	0.0033	0.00019	0.0088	0.00040	0.0077
3	0.0189	0.00022	0.0034	0.00020	0.0089	0.00047	0.0078
4	0.0190	0.00030	0.0035	0.00048	0.0090	0.00050	0.0079
5	0.0194	0.00030	0.0037	0.00050	0.0097	0.00060	0.0081
6	0.0196	0.00040	0.0039	0.00050	0.0097	0.00075	0.0083
7	0.0198	0.00050	0.0041	0.00060	0.0099	0.00086	0.0084
8	0.0203	0.00050	0.0041	0.00067	0.0101		0.0084
9	0.0208	0.00059	0.0041		0.0102		0.0095
10	0.0211	0.00060	0.0041		0.0103		
11	0.0218		0.0042		0.0103		
12	0.0237		0.0043		0.0107		
13			0.0044		0.0113		
<b>Mean</b>	<b>0.0201</b>	<b>(0.00037)</b>	<b>0.0039</b>	<b>(0.00041)</b>	<b>0.0098</b>	<b>(0.00054)</b>	<b>0.0082</b>
<b>Std Dev</b>	0.0016	-	0.0004	-	0.0009	-	0.0006
<b>C<sub>(95%)</sub></b>	0.0010	-	0.0003	-	0.0005	-	0.0004

Note: C<sub>(95%)</sub> 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  
Universal Scientific Laboratory Pty Ltd  
Genitest Inc  
Luo Yang Copper  
Shanghai Jinyi Test Technology  
TCR Engineering Services Ltd  
Raghavendra SpectroMet Laboratory  
Institute of Non-Ferrous Metals  
Tec-Eurolab  
AIM Metals and Alloys LP  
INCDMNR-IMNR  
Mineral & Metallurgical Laboratories  
Alpha Assembly Solutions  
AMG Superalloys UK Ltd  
Analyticka Laborator Lithea sro

Sheffield, England  
Milperra, NSW, Australia  
Montreal, Canada  
Luo Yang, He Nan, China  
Shanghai, China  
Mumbai, India  
Bangalore, India  
Gliwice, Poland  
Campogalliano, Italy  
Montreal, Canada  
Pantelimon, Romania  
Bangalore, India  
Altoona, PA, USA  
Rotherham, England  
Brno, Czech Republic

UKAS accreditation 0012  
NATA accreditation 0492  
PJ accreditation L17-153  
CNAL accreditation 0173  
CNAS accreditation 0041  
NABL accreditation 0367  
NABL accreditation T371  
PCA accreditation AB274  
ACCREDIA accreditation 52

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, 5, 7, 8, 11	-	-	2-4, 6, 10 9 volumetric (iodate) gravimetric (oxide)
Antimony	1, 3, 4, 6, 7, 9, 10, 12	12	2, 5, 8	11 volumetric (bromate)
Bismuth	1-3, 5, 8-10, 12, 14, 15	6	4, 11, 13	7 photometric (iodide)
Copper	2, 3, 6, 8-12, 14, 15	13	1, 4, 5, 7	
Arsenic	2, 4-9, 11-13	1	3, 10	
Iron	1, 2, 4, 6-9, 12-14	10	3, 5, 11	
Cadmium	1-4, 6-8, 10-12, 14, 15	15	9, 13	5 photometric
Silver	1, 2, 5, 7, 9, 10, 12		4, 6, 8, 11	3 volumetric (thiocyanate)
Zinc	5-10	1	2-4	
Nickel	1, 3, 4, 6, 9-12	5	2, 7, 8	13 photometric (dimethyl glyoxime)
Gold	1, 3-7	8	2	
Indium	1, 2, 5, 6, 8, 9, 11-13	3	4, 7, 10	
Tellurium	1-3, 5-7		4	
Mercury	1-6, 8, 9		7	

## Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO 17034 and the associated Guides, 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. 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. This certification will therefore expire in February 2039, 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.