

# CERTIFICATE OF ANALYSIS

## 95X BIS40P4 (batch A)

### Reference Material Information

Type: TIN/BISMUTH FUSIBLE ALLOY (CAST)  
Form and Size: Disc ~40mm diameter  
Produced by: MBH Analytical Ltd  
Certified and supplied by: MBH Analytical Ltd

### Assigned Values

#### Percentage element by weight

Element	Bi	Ag	Au	Al	As	Cd	Co	Cu	Fe
Value <sup>1</sup>	58.00	(0.70)	0.009	0.062	(0.007)	0.0092	0.0314	0.211	0.047
Uncertainty <sup>2</sup>	0.28	-	0.001	0.002	-	0.0005	0.0008	0.005	0.003

Element	Ge	Hg	In	Ni	Pb	Sb	Zn	Sn
Value <sup>1</sup>	(0.009)	0.047	2.02	0.073	0.210	0.144	0.0031	38.24
Uncertainty <sup>2</sup>	-	0.002	0.04	0.003	0.008	0.006	0.0008	0.28

Note: values given in parentheses are provided for information only.

### Definitions

- <sup>1</sup> The given values are the present best estimates of the true content for each element. Each value is based on the averaged results of a small interlaboratory testing programme, detailed on page 3.
- <sup>2</sup> The uncertainty values are estimates, 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.

### Validated by:

MBH ANALYTICAL LIMITED \_\_\_\_\_

on 17<sup>th</sup> January 2011

C Eveleigh



## **Method of Preparation**

This reference material was produced from commercial purity tin and bismuth; the trace elements were added as single elements or as binary alloys. 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 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 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 are method of use: Solders and similar alloys are generally prepared by machining on a lathe. However, users 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	Bi	Ag	Au	Al	As	Cd	Co	Cu	Fe
1	57.77	0.695	0.0088	0.0613	0.0045	0.0083	0.0306	0.206	0.0462
2	57.95	0.698	0.0091	0.0623	0.0078	0.0087	0.0308	0.208	0.0466
3	58.08		0.0092	0.0626	0.0089	0.0090	0.0309	0.209	0.0471
4	58.18		0.0094	0.0631		0.0092	0.0318	0.214	
5			0.0109			0.0094	0.0322	0.214	
6						0.0095	0.0322	0.215	
7						0.0101			
<b>Mean</b>	<b>58.00</b>	<b>(0.70)</b>	<b>0.0095</b>	<b>0.0623</b>	<b>(0.007)</b>	<b>0.0092</b>	<b>0.0314</b>	<b>0.211</b>	<b>0.0466</b>
<b>Std Dev</b>	0.18	-	0.0008	0.0008	-	0.0006	0.0007	0.004	0.0005
<b>C<sub>(95%)</sub></b>	0.28	-	0.0010	0.0012	-	0.0005	0.0008	0.004	0.0011

Sample	Ge	Hg	In	Ni	Pb	Sb	Zn	Sn
1	0.0072	0.0463	1.984	0.0721	0.194	0.141	0.0024	37.91
2	0.0100	0.0468	2.007	0.0724	0.207	0.143	0.0029	38.13
3	0.0103	0.0475	2.014	0.0729	0.208	0.144	0.0032	38.21
4			2.031	0.0731	0.209	0.146	0.0035	38.46
5			2.033	0.0743	0.213		0.0036	38.47
6			2.075	0.0752	0.220			
7					0.221			
<b>Mean</b>	<b>(0.009)</b>	<b>0.047</b>	<b>2.024</b>	<b>0.0733</b>	<b>0.210</b>	<b>0.144</b>	<b>0.0031</b>	<b>38.24</b>
<b>Std Dev</b>	-	0.001	0.031	0.0012	0.009	0.002	0.0005	0.24
<b>C<sub>(95%)</sub></b>	-	0.002	0.032	0.0012	0.008	0.003	0.0008	0.28

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

Universal Scientific Laboratory Pty Ltd  
Luo Yang Copper  
Institute of Iron & Steel Technology  
TCR Engineering Services Ltd  
Sargam Metals Pvt Ltd  
Raghavendra Spectrometallurgical Lab

Milperra, NSW, Australia  
Luo Yang, He Nan, China  
Shanghai, China  
Mumbai, India  
Chennai, India  
Bangalore, India

NATA accreditation 492  
CNAL accreditation 0173  
CNAL accreditation 0783  
NABL accreditation 0367  
NABL accreditation 0025

Note: to achieve the above accreditation (eg NATA, CNAL, 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
Bismuth	3	-	2 gravimetric 4 photometric (iodide) 1 volumetric (EDTA)
Silver	-	1, 2	
Gold	2, 4	1, 3	5 ICP-MS
Aluminium	2, 3	1, 4	
Arsenic	3	1, 2	
Cadmium	1, 2, 7	3, 4, 5, 6	
Cobalt	3, 6	1, 2, 4, 5	
Copper	2, 5	1, 3, 4	6 volumetric (thiosulfate)
Iron	3	1	2 photometric (orthophenanthroline)
Germanium	2	3	1 ICP-MS
Mercury	1, 3	2	
Indium	1, 2, 3	4, 5, 6	
Nickel	2, 6	1, 3, 4, 5	
Lead	1, 3, 4	2, 5, 6, 7	
Antimony	1, 2	3, 4	
Zinc	2, 5	3, 4	1 ICP-MS
Tin	-	-	1 gravimetric 3, 4, 5 volumetric (iodate) 2 photometric (phenyl fluorone)

## Notes

This Reference Material has been produced and certified, as far as 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 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 8mm. The rear portion of the disc, to a depth of ~7mm, 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 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 January 2031, although we reserve the right to make changes as issue revisions, in the intervening period.

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.