

# CERTIFICATE OF ANALYSIS

## 95X BIS40P1 (batch B)

### Reference Material Information

Type: TIN/BISMUTH FUSIBLE ALLOY (CAST)

Form and Size: Disc 40mm Diameter x 15mm Thickness

Produced by: MBH Analytical Ltd

Certified and supplied by: MBH Analytical Ltd

### Assigned Values

#### Percentage element by weight

Element	Sb	Sn	As	Cu	Bi
Value <sup>1</sup>	0.092	42.3	0.0101	0.0670	57.4
Uncertainty <sup>2</sup>	0.004	0.2	0.0013	0.0015	0.3

Element	Ag	Fe	Cd	In	Pb
Value <sup>1</sup>	0.035	(0.001)	0.0050	0.0164	0.043
Uncertainty <sup>2</sup>	0.003	-	0.0003	0.0013	0.003

Note: values given 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> May 2009

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## **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 - 2000, 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 a national authority. It is part of the 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 primary reference materials.

## **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 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	As	Cu	Bi
1	0.0879	41.85	0.0085	0.0645	57.07
2	0.0895	42.03	0.0085	0.0654	57.08
3	0.0899	42.09	0.0086	0.0658	57.20
4	0.0925	42.11	0.0095	0.0663	57.25
5	0.0926	42.30	0.0096	0.0675	57.61
6	0.0936	42.38	0.0098	0.0684	57.62
7	0.0940	42.44	0.0101	0.0684	57.70
8	0.0945	42.48	0.0102	0.0695	
9	0.096	42.54	0.0128		
10		42.57	0.0136		
<b>Mean</b>	<b>0.092</b>	<b>42.28</b>	<b>0.0101</b>	<b>0.0670</b>	<b>57.36</b>
<b>Std Dev</b>	0.003	0.24	0.0018	0.0017	0.27
<b>C<sub>(95%)</sub></b>	0.002	0.17	0.0013	0.0015	0.25

Sample	Ag	Fe	Cd	In	Pb
1	0.0300	0.0004	0.0043	0.0135	0.039
2	0.0301	0.0006	0.0045	0.0149	0.0409
3	0.0349	0.0008	0.0047	0.015	0.0411
4	0.035	0.0008	0.0050	0.0155	0.0423
5	0.0361	0.0011	0.0051	0.0159	0.0426
6	0.0365	0.0015	0.0052	0.0167	0.0433
7	0.0366	0.0016	0.0052	0.0175	0.0452
8	0.0375	0.0021	0.0054	0.0181	0.0480
9	0.0382	0.0024	0.0054	0.0182	
10			0.0056	0.019	
<b>Mean</b>	<b>0.035</b>	<b>(0.0013)</b>	<b>0.0050</b>	<b>0.0164</b>	<b>0.043</b>
<b>Std Dev</b>	0.003	-	0.0004	0.0018	0.003
<b>C<sub>(95%)</sub></b>	0.002	-	0.0003	0.0013	0.002

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 0012	Sheffield, England	UKAS accreditation
Bodycote Materials Testing Ltd	Middlesbrough, England	UKAS accreditation 0239
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 492
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
Raghavendra Spectrometallurgical Lab	Bangalore, India	
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Laboratory Inppamet	Calama, Chile	
Cookson Electronics, Fry Technologies	Altoona, PA, USA	

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
Antimony	1, 2, 4, 6, 7, 9	5, 8	3 volumetric (bromate)
Tin	1, 4, 6, 10	5	2, 7, 9 volumetric (periodate)
			3 gravimetric
			8 photometric (phenyl fluorone)
Arsenic	2, 3, 5, 9, 10	1, 6, 8	4, 7 photometric (molybdenum blue)
Copper	5, 6, 8	1-4, 7	
Bismuth	3-5	1	2, 7 volumetric (EDTA)
			6 gravimetric (dichromate)
Silver	2-4, 9	1, 5-8	
Iron	3-5, 7, 8	1, 2, 6	9 photometric (orthophenanthroline)
Cadmium	1, 2, 7, 9, 10	3-6, 8	
Indium	1, 3, 5-8, 10	2, 4, 9	
Lead	1, 3, 8	2, 4-7	

## Notes

This Reference Material has been produced and certified, as far as possible, in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, 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 10mm. The rear portion 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 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 May 2029, 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.