

CERTIFICATE OF ANALYSIS

95X PBS40P1 (batch A)

Reference Material Information

Type: TIN/LEAD/BISMUTH 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	Sb	Sn	As	Cu	Bi	Ag
Value ¹	0.016	42.6	0.005	0.025	13.8	0.011
Uncertainty ²	0.002	0.4	0.001	0.001	0.2	0.002

Element	Al	Fe	Zn	Cd	In	Pb
Value ¹	(0.0006)	(0.0006)	0.0010	0.0043	0.005	(43.6)
Uncertainty ²	-	-	0.0002	0.0002	0.001	-

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

Definitions

- ¹ 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.
- ² 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 _____
C Eveleigh

on 4th May 2011

Method of Preparation

This reference material was produced from commercial purity tin, lead 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 small 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 uncertainty for each element has initially been derived by combining these two factors, using the square-root of the summed squares. But the given uncertainty values have generally been rounded upwards due to the small scale of the analytical panel, and the lack of similar standards for cross-referencing.

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. In addition, some of the results derived as part of this testing programme will 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	Sb	Sn	As	Cu	Bi	Ag
1	0.0135	42.41	0.0027	0.0235	13.74	0.0089
2	0.0154	42.52	0.0038	0.0241	13.75	0.0105
3	0.0155	42.58	0.0045	0.0249	13.88	0.0109
4	0.0164	42.64	0.0046	0.0252	13.91	0.0112
5	0.0166	42.77	0.0055	0.0252	13.91	0.0113
6	0.0174		0.0057	0.0255		0.0135
Mean	0.0158	42.58	0.0047	0.0247	13.84	0.0111
Std Dev	0.0013	0.13	0.0011	0.0008	0.09	0.0015
C_(95%)	0.0014	0.17	0.0012	0.0008	0.10	0.0016

Sample	Al	Fe	Zn	Cd	In	Pb
1	0.0006	0.0002	0.0009	0.0039	0.0043	43.16
2	0.0006	0.0005	0.0009	0.0042	0.0048	43.70
3	0.0006	0.0005	0.0009	0.0043	0.0048	43.83
4	0.0006	0.0008	0.0010	0.0044	0.0051	
5	0.0007	0.0008	0.0014	0.0044	0.0052	
6				0.0045	0.0060	
Mean	0.0006	0.0006	0.0010	0.0043	0.0050	(43.56)
Std Dev	-	0.0003	0.0002	0.0002	0.0006	-
C_(95%)	-	0.0003	0.0002	0.0002	0.0006	-

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

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
Antimony	1, 2	3-6	
Tin	-	-	1 gravimetric 2, 4, 5 volumetric (iodate) 3 photometric (phenyl fluorone)
Arsenic	1, 2, 5	4, 6	3 photometric (molybdenum blue)
Copper	1, 3, 5	2, 6	4 volumetric (thiosulfate)
Bismuth	4	2	1 gravimetric 3 photometric (iodide) 5 volumetric (EDTA)
Silver	1, 3	2, 4-6	
Aluminium	4, 5	1, 2	3 ICP-MS
Iron	4, 5	1	2 photometric (orthophenanthroline) 3 ICP-MS
Zinc	2, 5	1, 3	4 ICP-MS
Cadmium	1, 4	2, 3, 5, 6	
Indium	2-4	1, 5, 6	
Lead	-	-	1-3 electrogravimetric

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.

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 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 May 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.