

# CERTIFICATE OF ANALYSIS

**91X S30PR3 (batch C)**

## Certified Reference Material Information

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

## Certified Analysis

### Percentage element by weight

Element	Sn	Sb	Bi	Cu	As	Ag
Value <sup>1</sup>	30.88	0.269	0.294	0.102	0.0126	0.024
Uncertainty <sup>2</sup>	0.10	0.010	0.007	0.002	0.0017	0.002

Element	Fe	Zn	Cd	Ni	In	Au
Value <sup>1</sup>	0.0016	(0.003)	0.0115	0.0269	0.0085	0.0063
Uncertainty <sup>2</sup>	0.0005	-	0.0005	0.0010	0.0010	0.0004

Note: information in parentheses is not certified - it is 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 9th January 2004  
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 was machined from the upper and lower surfaces of each disc, to minimise surface effects.

## **Sampling**

Samples for chemical analysis were taken from throughout the casting process. In addition approximately 10% of all discs, chosen at random from the complete cast, were checked for homogeneity.

## **Homogeneity**

The discs were checked for sample and batch uniformity using an optical emission spectrometer. One additional disc was checked for vertical uniformity using the same method.

For each of the surfaces checked, the differences between the averaged result and the overall mean value were evaluated to ensure that the overall homogeneity of the material comprising the batch satisfied the definition given in ISO guide 30 - 1992. However, the Zn shows signs of segregation.

Using the meaned data for each surface, standard deviation values were derived for each element. These values were combined with the 95% half-width confidence intervals ( $C_{(95\%)}$ ) obtained from the wet analysis programme, using the square-root of the summed squares, to derive the final uncertainty values.

The vertical uniformity check showed that this material is of satisfactory homogeneity for at least the first 10 mm of depth from the original chilled face.

## **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.

## **Traceability**

Most 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 method of use: Lead and tin 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.

When using OE, a minimum of three consistent replicate analyses is recommended to optimise precision and accuracy. 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	Ag
1	30.73	0.25	0.282	0.098	0.0098	0.020
2	30.83	0.255	0.282	0.099	0.010	0.0239
3	30.88	0.2568	0.292	0.099	0.0112	0.0239
4	30.89	0.259	0.295	0.0994	0.0126	0.0240
5	30.9	0.2646	0.2961	0.10	0.013	0.0242
6	30.91	0.273	0.298	0.1033	0.0141	0.0248
7	31.04	0.276	0.30	0.104	0.015	0.026
8		0.28	0.3031	0.1045	0.015	0.0289
9		0.287		0.105		
10		0.290		0.1077		
<b>Mean</b>	<b>30.88</b>	<b>0.269</b>	<b>0.294</b>	<b>0.102</b>	<b>0.0126</b>	<b>0.0244</b>
<b>Std Dev</b>	0.09	0.014	0.008	0.003	0.0021	0.0025
<b>C<sub>(95%)</sub></b>	0.09	0.010	0.007	0.002	0.0017	0.0021

Sample	Fe	Zn	Cd	Ni	In	Au
1	0.001	0.0020	0.0101	0.025	0.0066	0.0058
2	0.0014	0.002	0.011	0.0258	0.0069	0.0060
3	0.0015	0.0024	0.0111	0.026	0.0078	0.0063
4	0.0018	0.0027	0.0113	0.0261	0.008	0.0063
5	0.0019	0.0030	0.0116	0.0265	0.0083	0.0063
6	0.0020	0.0032	0.0117	0.027	0.0088	0.0067
7	<0.002	0.0041	0.012	0.0272	0.0097	0.0070
8	<0.005	0.0042	0.012	0.0275	0.0098	
9		0.0047	0.012	0.028	0.0106	
10			0.0124	0.030		
<b>Mean</b>	<b>0.0016</b>	<b>0.0031</b>	<b>0.0115</b>	<b>0.0269</b>	<b>0.0085</b>	<b>0.0063</b>
<b>Std Dev</b>	0.0004	0.0010	0.0007	0.0014	0.0014	0.0004
<b>C<sub>(95%)</sub></b>	0.0004	0.0008	0.0005	0.0010	0.0010	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

Birmingham Assay Office	Birmingham, England	UKAS accreditation 0667
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0014
Scientifics Ltd	Harwell, England	UKAS accreditation 0322
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing Inc	Hatfield, PA, USA	A2LA accreditation 0117
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Group Ltd	Luo Yang, He Nan, China	CNAL accreditation 0173
Maristas-Azterlan SL	Saibigain, Durango, Spain	ENAC accreditation 0059
Anglo-American Res. Laboratories Pty Ltd	Johannesburg, South Africa	
Cookson Electronics, Alpha-Fry Technologies	Altoona, PA, USA	
Genitest Inc	Montreal, Canada	

Note: to achieve National Accreditation (eg UKAS, NATA, A2LA, CNAL, ENAC), 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	1, 2, 4-6	-	3, 7 volumetric (iodate)
Antimony	1-6, 8-10	7	
Bismuth	1-3, 5-8	4	
Copper	1-6, 8-10	7	
Arsenic	1-4, 6-8	5	
Silver	1-4, 6-8	5	
Iron	1-4, 6-8	5	
Zinc	1-7, 9	8	
Cadmium	1-5, 7-10	6	
Nickel	1-5, 7-10	6	
Indium	1-4, 6-9	5	
Gold	1-5, 7	6	

## Notes

This Certified Reference Material has been produced and certified 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 ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The unidirectional solidification effects associated with semi-chill casting may lead 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. 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 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 2024, although we reserve the right to make changes as issue revisions, in the intervening period.

The Zn in this sample has segregated, and the value provided should be regarded as approximate only.

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.