

# CERTIFICATE OF ANALYSIS

**91X S63PR4 (batch A)**

## 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	Fe
Value <sup>1</sup>	66.8	0.093	0.030	0.021	<0.002	0.030	<0.005
Uncertainty <sup>2</sup>	0.5	0.005	0.001	0.001	-	0.001	-

Element	Zn	Cd	Ni	Au	Se	In	Te
Value <sup>1</sup>	<0.001	0.021	<0.005	0.05	<0.005	0.014	0.006
Uncertainty <sup>2</sup>	-	0.001	-	0.01	-	0.003	0.002

## 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 14th December 2001



## **Method of Preparation**

This reference material was produced from commercial-purity lead and tin, pure elements, binaries and master alloys. The melt was cast by sequential transfer of aliquots into steel chill moulds. 2mm has been removed from the cast face to minimise any surface effects.

## **Sampling**

Samples for chemical analysis, and discs for homogeneity checks, were taken from various positions within the casting process.

## **Homogeneity**

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. One additional disc was checked for vertical uniformity using the same method.

Multiple measurements were taken from each surface under test.

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.

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.

Using the individual data from each check, 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.

## **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 ISO Guide 25-1990, using documented standard reference methods and validated by appropriate reference materials.

The individual values listed overpage are the average of each analyst's results.

## **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	Fe
1	66.68	0.09	0.029	0.019	0.001	0.029	0.001
2	66.8	0.09	0.030	0.019	0.002	0.029	0.002
3	66.8	0.091	0.030	0.020	<0.002	0.030	0.003
4	66.9	0.091	0.030	0.020	<0.002	0.03	0.004
5	67.0	0.092	0.031	0.020	<0.002	0.030	<0.005
6		0.100		0.021		0.031	<0.005
7		0.10		0.022			
8				0.023			
<b>Mean</b>	<b>66.84</b>	<b>0.093</b>	<b>0.030</b>	<b>0.021</b>	<b>&lt;0.002</b>	<b>0.030</b>	<b>&lt;0.005</b>
<b>Std Dev</b>	0.12	0.005	0.001	0.001	-	0.001	-
<b>C<sub>(95%)</sub></b>	0.15	0.004	0.001	0.001	-	0.001	-

Sample	Zn	Cd	Ni	Au	Se	In	Te
1	<0.0001	0.019	0.001	0.035	<0.001	0.011	0.003
2	0.001	0.020	0.002	0.038	<0.002	0.014	0.006
3	0.001	0.02	0.002	0.048	0.002	0.015	0.006
4	0.001	0.021	0.003	0.057	0.003	0.015	0.006
5	<0.001	0.021	0.003	0.059	0.004		0.007
6	<0.001	0.021	<0.001		0.004		
7		0.022	<0.002				
<b>Mean</b>	<b>&lt;0.001</b>	<b>0.021</b>	<b>&lt;0.005</b>	<b>0.047</b>	<b>&lt;0.005</b>	<b>0.014</b>	<b>0.006</b>
<b>Std Dev</b>	-	0.001	-	0.011	-	0.002	0.002
<b>C<sub>(95%)</sub></b>	-	0.001	-	0.013	-	0.003	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

RoTech Laboratories Ltd  
Metals Technology Testing Ltd  
JB Elds Ltd  
London & Scandinavian Met Co  
Bodycote Materials Testing Ltd  
Coleshill Laboratories Ltd  
British Lead Mills Ltd  
Analytichem Consultants Ltd

Wednesbury, England  
Sheffield, England  
Stoke, England  
Rotherham, England  
Middlesbrough  
Coleshill, England  
Welwyn, England  
Solihull, England

NAMAS accreditation 0366  
NAMAS accreditation 0963  
NAMAS accreditation 1173  
NAMAS accreditation 1091  
NAMAS accreditation 0239

## Analytical Methods Used

ELEMENT	RESULT No. & METHOD		
	ICP-AES	FAAS	OTHER
Tin	5	2, 3, 4	1 volumetric (iodate)
Antimony	1, 3	2, 4, 5, 6, 7	
Bismuth	1, 2	3, 4, 5	
Copper	2, 3, 8	1, 4, 5, 6, 7	
Arsenic	1, 2, 3	4, 5	
Silver	1, 3	2, 4, 5, 6	
Iron	1, 4	2, 3, 5, 6	
Zinc	2, 6	1, 3, 4, 5	
Cadmium	4, 5	1, 2, 3, 6, 7	
Nickel	2, 6	1, 3, 4, 5, 7	
Gold	1, 2, 3	4, 5	
Selenium	1, 2, 3	4, 5, 6	
Indium	1, 4	2, 3	
Tellurium	1, 2	3, 4, 5	

## 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.

## Notes

This Certified Reference Material was originally analysed and certified in October 1999. The certification has been upissued for clarity, and to indicate additional uncertainty and traceability information. This revised certificate is in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, and wherever possible takes into account the requirements of ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

Note: to achieve accreditation to NAMAS (UK National Measurement Accreditation Scheme), test houses were required to demonstrate conformity to the general requirements of ISO Guide 25 and ISO9002.

The combination of alloying elements used in a complex material of this type, coupled with the unidirectional solidification effects associated with 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. 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 original analysis. This certification will therefore expire in October 2019, although we reserve the right to make changes as issue revisions, in the intervening period.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.