

# CERTIFICATE OF ANALYSIS

**73X SC9 (batch B)**

## Certified Reference Material Information

Type: TIN WHITE METAL (CAST)  
Form and Size: Disc ~40mm diameter  
Manufactured by: MBH Analytical Ltd  
Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	Sb	As	Bi	Pb	Cu	Fe
Value <sup>1</sup>	8.31	0.117	0.0825	0.305	8.03	0.053
Uncertainty <sup>2</sup>	0.05	0.004	0.0016	0.005	0.05	0.002

Element	Ni	Al	Cd	In	Ag	Co
Value <sup>1</sup>	0.0084	(0.0010)	0.0506	0.0059	0.0055	0.0051
Uncertainty <sup>2</sup>	0.0006	-	0.0007	0.0003	0.0004	0.0001

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:

on 3rd December 2016

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

C Eveleigh

## **Method of Preparation**

This reference material was produced from commercial-purity tin, with the major and trace elements added as single elements or as binary 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**

Milled samples for chemical analysis were taken from several positions within the batch. In addition, at least 15% of all discs were selected for homogeneity checking.

## **Homogeneity**

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Multiple measurements were taken from each surface under test.

Using the meaned data from 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 turnings taken from samples representative of the product. It was performed by participating laboratories mostly operating within the terms of EN ISO/IEC 17025 - 2005, using documented standard methods of analysis.

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 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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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 method of use: Tin is 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	As	Bi	Pb	Cu	Fe
1	8.261	0.1060	0.0798	0.2920	7.999	0.0495
2	8.270	0.1070	0.0800	0.2964	8.000	0.0496
3	8.270	0.1102	0.0802	0.2994	8.005	0.0499
4	8.290	0.1110	0.0802	0.2998	8.010	0.0511
5	8.297	0.1115	0.0804	0.3010	8.022	0.0516
6	8.301	0.1188	0.0807	0.3031	8.022	0.0518
7	8.308	0.1190	0.0812	0.3047	8.030	0.0524
8	8.318	0.1200	0.0812	0.3061	8.036	0.0524
9	8.331	0.1209	0.0814	0.3067	8.045	0.0524
10	8.334	0.1238	0.0838	0.3076	8.048	0.0529
11	8.334	0.1246	0.0841	0.3106	8.056	0.0534
12	8.369	0.1252	0.0844	0.3110	8.100	0.0537
13	8.372		0.0851	0.3115		0.0537
14			0.0867	0.3136		0.0547
15			0.0882	0.3140		0.0616
<b>Mean</b>	<b>8.312</b>	<b>0.1165</b>	<b>0.0825</b>	<b>0.3052</b>	<b>8.031</b>	<b>0.0527</b>
<b>Std Dev</b>	0.036	0.0070	0.0027	0.0065	0.029	0.0029
<b>C<sub>(95%)</sub></b>	0.021	0.0044	0.0015	0.0036	0.018	0.0016

Sample	Ni	Al	Cd	In	Ag	Co
1	0.0068	0.0006	0.0494	0.0049	0.0047	0.0048
2	0.0070	0.0006	0.0495	0.0052	0.0049	0.0048
3	0.0074	0.0007	0.0496	0.0053	0.0050	0.0049
4	0.0075	0.0009	0.0496	0.0056	0.0051	0.0049
5	0.0077	0.0009	0.0496	0.0058	0.0051	0.0050
6	0.0078	0.0010	0.0498	0.0059	0.0054	0.0050
7	0.0079	0.0011	0.0500	0.0060	0.0054	0.0050
8	0.0082	0.0011	0.0502	0.0063	0.0055	0.0051
9	0.0084	0.0011	0.0506	0.0063	0.0057	0.0051
10	0.0084	0.0012	0.0506	0.0064	0.0058	0.0052
11	0.0086	0.0013	0.0509	0.0065	0.0064	0.0053
12	0.0090		0.0511	0.0066	0.0068	0.0053
13	0.0093		0.0511			0.0053
14	0.0098		0.0514			0.0054
15	0.0102		0.0523			0.0056
16	0.0103		0.0531			
<b>Mean</b>	<b>0.0084</b>	<b>0.0009</b>	<b>0.0506</b>	<b>0.0059</b>	<b>0.0055</b>	<b>0.0051</b>
<b>Std Dev</b>	0.0011	0.0002	0.0011	0.0005	0.0006	0.0002
<b>C<sub>(95%)</sub></b>	0.0006	0.0002	0.0006	0.0003	0.0004	0.0001

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

Exova Ltd	Middlesbrough, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
Universal Scientific Laboratory Pty Ltd	Milperra, Australia	NATA accreditation	0492
Genitest Inc.	Montreal, QC, Canada	PRI accreditation	123077
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation	0173
Bureau Veritas CPS Ltd	Chennai, India	NABL accreditation	0025
TCR Engineering Services PVT. Ltd	Mumbai India	NABL accreditation	0367
Raghavendra Spectro Metallurgical Laboratory	Bangalore, India	NABL accreditation	0371
Institute of Non-ferrous Metals	Gliwice, Poland	PCA accreditation	AB274
TEC Eurolab SRL	Modena, Italy	ACCREDIA accreditation	52
Mineral and Metallurgical Laboratories	Bangalore, India		
AIM Metals and Alloys LP	Montreal, QC, Canada		
AMG Superalloys UK Ltd	Rotherham, UK		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
Laboratory Inppamet	Calama, Chile		
Coleshill Laboratories Ltd	Coleshill, UK		

Note: to achieve the above accreditation (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, 3, 6, 8, 10, 12	4, 7, 11, 13	2, 5, 9	volumetric (bromate)
Arsenic	3-12	1	2	photometric (molybdenum blue)
Bismuth	1-3, 5-7, 9, 11-13	8, 10, 14, 15	4	photometric (thiourea)
Lead	3-7, 9, 11, 13, 14	1, 2, 8, 10, 15	12	electrogravimetric
Copper	1, 4, 6-8, 11	2, 9-10	3, 5	volumetric (thiosulfate)
			12	electrogravimetric
Iron	1, 3, 7, 9-11, 13-15	2, 5, 6, 8, 12	4	volumetric (Redox)
Nickel	2, 4-6, 8, 10-15	1, 3, 7, 9	15	ICP-MS
Aluminium	1-3, 6-9, 11	4, 5, 10		
Cadmium	1, 2, 6, 8-10, 12-14, 16	3-5, 7, 11, 15		
Indium	1-5, 7, 9-12	6, 8		
Silver	1-3, 5-7, 9, 12	4, 8, 11	10	ICP-MS
Cobalt	2, 4, 5, 7-11, 14, 15	1, 3, 6, 12, 13		

## Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2015 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 this method of 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 for a depth of 7mm. Material to the rear of the disc, to a depth of ~5 mm, is not certified.

This material is liable to superficial corrosion. There is also a possibility for microstructural changes due to recrystallisation, and diffusion effects may lead to the concentration of some elements at the surface. For X-ray and other superficial sampling techniques, it is therefore recommended that the surface is refreshed immediately prior to use. In all other respects, this sample will 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. Technical support for this certification will therefore expire in December 2036, although we reserve the right to make changes as issue revisions, in the intervening period.

This material is also available in the form of chippings

The manufacture, analysis and certification of this product were supervised by L Maxim, Technical Director, MBH Analytical Ltd.

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