

# CERTIFICATE OF ANALYSIS

**73X SC7 (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	Ni	Ag
Value <sup>1</sup>	13.86	0.059	0.013	0.32	5.71	0.0160	0.0059
Uncertainty <sup>2</sup>	0.10	0.004	0.002	0.01	0.04	0.0008	0.0005

Element	Fe	Al	Cd	Zn	In	Co	S
Value <sup>1</sup>	(0.043)	(0.0009)	0.0019	0.0033	0.0194	0.0143	(0.0007)
Uncertainty <sup>2</sup>	-	-	0.0001	0.0005	0.0010	0.0006	-

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 23rd December 2012

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

C Eveleigh

## **Method of Preparation**

This reference material was produced from commercial tin; the major alloys and traces were added as single elements or as master alloys. The melt was cast by sequential transfer of aliquots into iron moulds. At least 1mm 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 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. In addition, some of the results derived as part of this testing programme 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 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	Ni	Ag
1	13.73	0.0546	0.0094	0.302	5.654	0.0154	0.0046
2	13.75	0.0550	0.0098	0.303	5.664	0.0154	0.0049
3	13.75	0.0559	0.0098	0.311	5.679	0.0155	0.0053
4	13.75	0.0561	0.0104	0.314	5.694	0.0156	0.0058
5	13.78	0.0564	0.0110	0.315	5.706	0.0157	0.0059
6	13.85	0.0568	0.0112	0.318	5.708	0.0157	0.0059
7	13.90	0.0568	0.0118	0.319	5.717	0.0159	0.0060
8	13.96	0.0617	0.0132	0.320	5.720	0.0161	0.0060
9	13.97	0.0664	0.0145	0.321	5.724	0.0161	0.0064
10	14.00	0.0669	0.0148	0.322	5.759	0.0161	0.0065
11	14.01	0.0674	0.0152	0.335	5.772	0.0170	0.0070
12			0.0155	0.335		0.0170	0.0070
13			0.0177	0.338			
14			0.0179	0.339			
<b>Mean</b>	<b>13.86</b>	<b>0.0594</b>	<b>0.0130</b>	<b>0.321</b>	<b>5.709</b>	<b>0.0160</b>	<b>0.0059</b>
<b>Std Dev</b>	0.11	0.0051	0.0030	0.012	0.036	0.0006	0.0007
<b>C<sub>(95%)</sub></b>	0.08	0.0034	0.0017	0.007	0.024	0.0003	0.0005

Sample	Fe	Al	Cd	Zn	In	Co	S
1	0.0365	0.0004	0.0015	0.0027	0.0173	0.0124	0.0003
2	0.0383	0.0005	0.0015	0.0027	0.0176	0.0131	0.00058
3	0.0400	0.0008	0.0018	0.0028	0.0185	0.0134	0.00082
4	0.0406	0.0009	0.0018	0.0029	0.0191	0.0136	0.00083
5	0.0423	0.0010	0.0019	0.0033	0.0200	0.0138	0.0009
6	0.0433	0.0011	0.0019	0.0035	0.0203	0.0139	0.0010
7	0.0433	0.0011	0.0019	0.0036	0.0203	0.0144	
8	0.0445	0.0012	0.0020	0.0042	0.0204	0.0144	
9	0.0452		0.0020	0.0042	0.0209	0.0147	
10	0.0474		0.0020			0.0147	
11	0.0489		0.0021			0.0148	
12			0.0021			0.0151	
13			0.0022			0.0151	
14			0.0022			0.0162	
<b>Mean</b>	<b>0.0428</b>	<b>0.0009</b>	<b>0.0019</b>	<b>0.0033</b>	<b>0.0194</b>	<b>0.0143</b>	<b>0.00074</b>
<b>Std Dev</b>	0.0037	0.0003	0.0002	0.0006	0.0013	0.0010	0.00026
<b>C<sub>(95%)</sub></b>	0.0025	0.0002	0.0001	0.0005	0.0010	0.0006	0.00027

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

Exova Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
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 Laboratory Pvt Ltd	Chennai, India	NABL accreditation 0025
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
AIM Metals and Alloys LP	Montreal, Canada	
London & Scandinavian Met Co	Rotherham, England	
Coleshill Laboratories Ltd	Coleshill, England	
Raghavendra Spectrometallurgical Laboratory	Bangalore, India	
De Bruyn Spectroscopic Solutions	Johannesburg, South Africa	

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	1-4, 7, 8, 10	6, 9, 11	5 volumetric (bromate)
Arsenic	2-4, 6, 7, 9-11	1, 5	8 photometric (molybdenum blue)
Bismuth	2, 3, 5-7, 9-11, 13, 14	1, 8, 12	4 photometric (iodide)
Lead	2, 3, 5-7, 10-14	1, 4, 8, 9	
Copper	1, 2, 4, 6, 7, 10, 11	3, 8, 9	5 volumetric (thiosulfate)
Nickel	1-3, 6-10	4, 5, 11, 12	
Silver	1, 2, 4-6, 9-11	3, 7, 8, 12	
Iron	1, 4, 6, 7, 9-11	2, 5, 8	3 photometric (orthophenanthroline)
Aluminium	3-5, 7, 8	1, 6	2 photometric (chrome azurol S)
Cadmium	1-3, 5-9, 12-14	4, 10, 11	
Zinc	1, 2, 4-6, 8, 9	3, 7	
Indium	1, 2, 4-6, 8, 9	3, 7	
Cobalt	1, 2, 4-6, 8-10, 13, 14	3, 7, 11, 12	
Sulfur	4, 5	-	1-3, 6 combustion ( infra-red detection)

## Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 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 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. 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. This certification will therefore expire in December 2032, 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.