MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

73X SC11 (batch C)

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	Cu	Ag	Pb	Sb	Bi	Ni
Value [1]	**11.51**	**(0.056)**	**0.0630**	**12.98**	**0.554**	**0.110**
Uncertainty [2]	0.05	-	0.0013	0.07	0.005	0.003

Element	Cd	Zn	Fe	As	P
Value [1]	**1.72**	**0.005**	**0.0052**	**0.248**	**0.025**
Uncertainty [2]	0.02	0.001	0.0006	0.004	0.001

Note: values given in parentheses are not certified - they are provided for information only.

Definitions

[1] 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.

[2] 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 tin; the major and trace elements were added as single elements or as master alloys. The melt was cast by sequential transfer of aliquots into iron moulds. 2mm has been removed from the working face of each disc, 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	Cu	Ag	Pb	Sb	Bi	Ni
1	11.462	0.0522	0.0598	12.910	0.5468	0.1001
2	11.480	0.0523	0.0601	12.920	0.5469	0.1004
3	11.493	0.0536	0.0602	12.923	0.5474	0.1020
4	11.494	0.0548	0.0609	12.970	0.5484	0.1060
5	11.500	0.0573	0.0612	12.974	0.5494	0.1063
6	11.504	0.0575	0.0615	12.993	0.5498	0.1066
7	11.505	0.0579	0.0621	13.010	0.5500	0.1081
8	11.522	0.0583	0.0622	13.010	0.5503	0.1081
9	11.530	0.0588	0.0624	13.030	0.5512	0.1101
10	11.544	0.0602	0.0629	13.036	0.5520	0.1102
11	11.552		0.0638		0.5542	0.1104
12	11.570		0.0640		0.5590	0.1138
13			0.0645		0.5620	0.1160
14			0.0646		0.5626	0.1163
15			0.0648		0.5630	0.1164
16			0.0661		0.5654	0.1170
17			0.0667			0.1179
18			0.0671			0.1210
Mean	**11.513**	**0.0563**	**0.0630**	**12.978**	**0.5536**	**0.1104**
Std Dev	0.031	0.0028	0.0023	0.046	0.0065	0.0063
$C_{(95\%)}$	0.020	0.0020	0.0011	0.033	0.0035	0.0031

Sample	Cd	Zn	Fe	As	P
1	1.6762	0.0047	0.0046	0.2412	0.0245
2	1.6820	0.0047	0.0049	0.2430	0.0247
3	1.6880	0.0049	0.0050	0.2445	0.0248
4	1.7000	0.0050	0.0050	0.2448	0.0252
5	1.7010	0.0051	0.0051	0.2461	0.0253
6	1.7030	0.0052	0.0051	0.2463	0.0253
7	1.7076	0.0053	0.0052	0.2469	0.0255
8	1.7080	0.0054	0.0053	0.2476	0.0257
9	1.7200	0.0056	0.0053	0.2487	0.0261
10	1.7333	0.0056	0.0054	0.2505	0.0262
11	1.7370	0.0057	0.0054	0.2520	0.0263
12	1.7516	0.0058	0.0058	0.2529	
13	1.7530	0.0061		0.2530	
14	1.7582	0.0064			
15	1.7591				
Mean	**1.7185**	**0.0054**	**0.0052**	**0.2475**	**0.0254**
Std Dev	0.0283	0.0005	0.0003	0.0038	0.0006
$C_{(95\%)}$	0.0157	0.0003	0.0002	0.0023	0.0004

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, 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, 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
Copper	2-4, 6, 10-12	1, 5, 7, 9	8	volumetric (thiosulfate)
Silver	1-4, 7, 8	5, 6, 9, 10		
Lead	1, 3-5, 9-12, 14, 15, 18	6-8, 13, 16, 17	2	electrogravimetric
Antimony	2, 4, 6, 8, 10	3, 9	1, 5, 7	volumetric (bromate)
Bismuth	2-4, 6-9, 11, 14-16	1, 5, 12-13	10	photometric (thiourea)
Nickel	2, 5, 6, 8, 11-17	1, 4, 7, 9, 10, 18	3	gravimetric (dimethyl glyoxime)
Cadmium	1-3, 5, 7, 10, 12-14	4, 6, 9, 11, 15	8	gravimetric (o-hydroxyphenylbenz.
Zinc	5-8, 10-13	2-4, 9, 14	1	ICP-MS
Iron	2, 4, 6, 7, 9, 11, 12	1, 3, 5, 10	8	ICP-MS
Arsenic	1-4, 6-12	-	5	ICP-MS
			13	photometric (molybdenum blue)
Phosphorus	1-4, 6-9	-	5, 11	volumetric (alkalimetric)
			10	ICP-MS

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 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.