MBH ANALYTICAL LTD

HOLLAND HOUSE • QUEENS ROAD • BARNET • EN5 4DJ • ENGLAND • TEL: +44 (0)20 8441 2024 • FAX: +44 (0)20 8449 0810

email: info@mbh.co.uk web: www.mbh.co.uk

CERTIFICATE OF ANALYSIS

74X CA4 (batch D)

Certified Reference Material Information

Type:	TIN-BASE LEAD-FREE SOLDER (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	Ag	Cu	Pb	Bi	Sb	Fe	As	Co
Value [1]	**3.07**	**0.552**	**0.081**	**0.0515**	**0.0612**	**0.0040**	**0.0030**	**0.0005**
Uncertainty [2]	0.05	0.007	0.003	0.0011	0.0017	0.0003	0.0003	0.0001

Element	Au	Cd	Zn	Ni	Cr	In	Hg
Value [1]	**0.0020**	**0.0025**	**0.0051**	**0.0589**	**0.0010**	**0.009**	**0.0075**
Uncertainty [2]	0.0001	0.0001	0.0005	0.0012	0.0002	0.001	0.0006

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 10th January 2019

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 individual 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. Approximately 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 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	Ag	Cu	Pb	Bi	Sb	Fe	As	Co
1	2.995	0.5440	0.0749	0.0477	0.0574	0.0036	0.0023	0.00025
2	2.998	0.5470	0.0767	0.0488	0.0582	0.0037	0.0025	0.00036
3	3.004	0.5472	0.0773	0.0503	0.0585	0.0037	0.0028	0.00038
4	3.013	0.5472	0.0791	0.0504	0.0587	0.0038	0.0028	0.00044
5	3.031	0.5490	0.0792	0.0505	0.0613	0.0040	0.0029	0.00050
6	3.077	0.5529	0.0800	0.0508	0.0618	0.0040	0.0030	0.00060
7	3.078	0.5530	0.0823	0.0510	0.0624	0.0041	0.0032	0.00065
8	3.100	0.5533	0.0829	0.0519	0.0632	0.0041	0.0032	0.00070
9	3.125	0.5562	0.0830	0.0520	0.0633	0.0043	0.0033	0.00070
10	3.131	0.5660	0.0839	0.0526	0.0638	0.0043	0.0038	0.00080
11	3.150		0.0866	0.0529	0.0643	0.0044		
12	3.164			0.0534				
13				0.0540				
14				0.0544				
Mean	**3.072**	**0.5516**	**0.0805**	**0.0515**	**0.0612**	**0.0040**	**0.0030**	**0.00054**
Std Dev	0.062	0.0063	0.0035	0.0019	0.0025	0.0003	0.0004	0.00018
$C_{(95\%)}$	0.040	0.0045	0.0024	0.0011	0.0017	0.0002	0.0003	0.00013

Sample	Au	Cd	Zn	Ni	Cr	In	Hg
1	0.0017	0.0024	0.0045	0.0555	0.0007	0.0065	0.0062
2	0.0017	0.0024	0.0045	0.0568	0.0007	0.0071	0.0065
3	0.0019	0.0024	0.0047	0.0571	0.0008	0.0075	0.0071
4	0.0019	0.0024	0.0048	0.0575	0.0009	0.0080	0.0073
5	0.0020	0.0025	0.0048	0.0585	0.0009	0.0082	0.0074
6	0.0020	0.0025	0.0050	0.0586	0.0010	0.0092	0.0077
7	0.0020	0.0025	0.0050	0.0587	0.0010	0.0094	0.0079
8	0.0020	0.0026	0.0051	0.0589	0.0011	0.0103	0.0079
9	0.0021	0.0026	0.0051	0.0591	0.0015	0.0104	0.0082
10	0.0021	0.0026	0.0054	0.0600	0.0016	0.0104	0.0092
11	0.0021	0.0027	0.0058	0.0606		0.0106	
12	0.0024		0.0060	0.0608		0.0112	
13	0.0024			0.0609		0.0114	
14				0.0612		0.0115	
Mean	**0.0020**	**0.0025**	**0.0051**	**0.0589**	**0.0010**	**0.0094**	**0.0075**
Std Dev	0.0002	0.0001	0.0005	0.0017	0.0003	0.0017	0.0009
$C_{(95\%)}$	0.0001	0.0001	0.0003	0.0010	0.0002	0.0010	0.0006

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

Element Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Anchorcert Analytical	Birmingham, England	UKAS accreditation 0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest Inc	Montreal, Canada	PJ accreditation L17-153
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
AIM Metals and Alloys LP	Montreal, Canada	
INCDMNR-IMNR	Pantelimon, Romania	
Mineral & Metallurgical Laboratories	Bangalore, India	
Alpha Assembly Solutions	Altoona, PA, USA	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

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	ICP-MS	FAAS		OTHER
Silver	2-4, 6, 11, 12	-	1, 5, 9, 10	7, 8	volumetric (thiocyanate)
Copper	2, 4, 6-9	-	3, 5, 10	1	volumetric (thiosulfate)
Lead	1, 2, 4, 6-9, 11	10	3, 5		
Bismuth	1, 3, 5, 6, 8-13	-	4, 7, 14	2	gravimetric
Antimony	2-5, 8-11	-	6, 7	1	volumetric (bromate)
Iron	1, 3, 4, 6-8, 11	10	2, 5, 9		
Arsenic	2-8, 10	9	1		
Cobalt	1, 2, 5-10	4	3		
Gold	2-4, 6, 7, 10-13	5	1, 8, 9		
Cadmium	2-7, 9, 10	11	1, 8		
Zinc	2, 3, 5-12	-	1, 4		
Nickel	1-4, 6, 8, 10-14	-	5, 7, 9		
Chromium	1, 2, 5, 7, 8, 10	4	3, 6, 9		
Indium	1-4, 7, 9-11, 13, 14	6	5, 8, 12		
Mercury	1-4, 6-10	-	5		

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO 17034 and the associated Guides, 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 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 January 2039, 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.