

CERTIFICATE OF ANALYSIS

74X CA8 (batch B)

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	P	Ge
Value ¹	2.47	0.950	0.084	0.0172	0.0180	0.010	0.0020
Uncertainty ²	0.03	0.010	0.002	0.0006	0.0012	0.002	0.0005

Element	Fe	As	In	Cd	Ni	Co	Hg
Value ¹	0.0043	0.0144	0.0062	0.0101	0.0020	0.0202	0.101
Uncertainty ²	0.0005	0.0006	0.0004	0.0002	0.0001	0.0006	0.003

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:

MBH ANALYTICAL LIMITED _____

on 30th September 2011

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. 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	Ag	Cu	Pb	Bi	Sb	P	Ge
1	2.419	0.944	0.0798	0.0157	0.0150	0.0080	0.0015
2	2.426	0.946	0.0801	0.0159	0.0161	0.0082	0.0017
3	2.441	0.947	0.0806	0.0163	0.0162	0.0092	0.0018
4	2.443	0.947	0.0810	0.0166	0.0166	0.0094	0.0019
5	2.451	0.948	0.0826	0.0167	0.0168	0.0105	0.0019
6	2.453	0.949	0.0837	0.0167	0.0170	0.0115	0.0023
7	2.461	0.950	0.0842	0.0168	0.0175	0.0116	0.0026
8	2.483	0.951	0.0845	0.0169	0.0178	0.0122	
9	2.496	0.952	0.0849	0.0172	0.0181		
10	2.496	0.953	0.0853	0.0172	0.0182		
11	2.500	0.954	0.0855	0.0176	0.0191		
12	2.500	0.960	0.0855	0.0179	0.0195		
13	2.505		0.0861	0.0186	0.0197		
14	2.506		0.0878	0.0190	0.0198		
15			0.0885	0.0190	0.0204		
16			0.0891		0.0205		
Mean	2.470	0.950	0.0843	0.0172	0.0180	0.0101	0.0020
Std Dev	0.031	0.004	0.0029	0.0010	0.0017	0.0016	0.0004
C_(95%)	0.018	0.003	0.0015	0.0006	0.0009	0.0013	0.0003

Sample	Fe	As	In	Cd	Ni	Co	Hg
1	0.0033	0.0133	0.0052	0.0096	0.0015	0.0183	0.0940
2	0.0035	0.0136	0.0055	0.0097	0.0016	0.0184	0.0957
3	0.0035	0.0137	0.0057	0.0097	0.0018	0.0186	0.0985
4	0.0039	0.0139	0.0057	0.0097	0.0018	0.0193	0.0987
5	0.0039	0.0143	0.0061	0.0098	0.0019	0.0195	0.0988
6	0.0040	0.0143	0.0062	0.0099	0.0019	0.0201	0.1026
7	0.0041	0.0144	0.0062	0.0099	0.0019	0.0201	0.1030
8	0.0041	0.0146	0.0063	0.0101	0.0020	0.0205	0.1053
9	0.0041	0.0147	0.0063	0.0101	0.0020	0.0205	0.1054
10	0.0043	0.0147	0.0064	0.0102	0.0021	0.0206	0.1064
11	0.0044	0.0150	0.0065	0.0103	0.0021	0.0207	0.1070
12	0.0048	0.0159	0.0065	0.0104	0.0021	0.0207	
13	0.0050		0.0067	0.0104	0.0023	0.0209	
14	0.0050		0.0072	0.0105	0.0023	0.0211	
15	0.0050			0.0107	0.0025	0.0222	
16	0.0052					0.0223	
Mean	0.0043	0.0144	0.0062	0.0101	0.0020	0.0202	0.1014
Std Dev	0.0006	0.0007	0.0005	0.0003	0.0003	0.0012	0.0045
C_(95%)	0.0003	0.0004	0.0003	0.0002	0.0001	0.0006	0.0030

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 Sheffield Assay Office Laboratory Testing, Inc Genitest, Inc Universal Scientific Laboratory Pty Ltd Luo Yang Copper Institute of Iron & Steel Technology TCR Engineering Services Ltd Sargam Metals Pvt Ltd Institute of Non-Ferrous Metals AIM Metals and Alloys LP De Bruyn Spectroscopic Solutions Ltd Raghavendra Spectrometallurgical Laboratory London & Scandinavian Met Co Laboratory Inppamet Coleshill Laboratories Ltd	Middlesbrough, England Sheffield, England Hatfield, PA, USA Montreal, Canada Milperra, NSW, Australia Luo Yang, He Nan, China Shanghai, China Mumbai, India Chennai, India Gliwice, Poland Montreal, Canada Johannesburg, South Africa Bangalore, India Rotherham, England Calama, Chile Birmingham, England	UKAS accreditation 0239 UKAS accreditation 0012 A2LA accreditation 0114 PRI accreditation 123077 NATA accreditation 0492 CNAL accreditation 0173 CNAL accreditation 0783 NABL accreditation 0367 NABL accreditation 0025 PCA accreditation AB274
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Note: to achieve the above-noted 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
Silver	4, 5, 8, 10-12	1, 3, 6, 7, 13, 14	2, 9 volumetric (thiocyanate)
Copper	1, 6, 8-12	2-4, 7	5 volumetric (thiosulfate)
Lead	2, 3, 5-8, 10, 12, 13, 15, 16	1, 4, 9, 11, 14	
Bismuth	2, 3, 5, 6, 8, 10-14	1, 4, 7, 15	9 photometric (iodide)
Antimony	1, 2, 4, 5, 7, 9, 10, 12-14, 16	3, 6, 8, 11	15 volumetric (bromate)
Phosphorus	1, 3, 5-7	-	2, 4 photometric (molybdenum blue)
Germanium	2-4, 6, 7	5	8 ICP-MS
Iron	1-4, 6, 8-10, 13, 15, 16	5, 7, 11, 12, 14	1 ICP-MS
Arsenic	1-3, 6, 7, 9-12	4, 5	8 photometric (molybdenum blue)
Indium	1-4, 6-8, 10-12, 14	5, 9, 13	
Cadmium	1, 3-6, 9-14	2, 7, 8, 15	
Nickel	1-3, 6-8, 11-13, 15	4, 5, 9, 10, 14	
Cobalt	1, 3, 5, 6, 9-11, 13, 15, 16	2, 4, 7, 8, 12, 14	
Mercury	1-4, 6-9	5, 10, 11	

Notes

This Certified Reference Material has been produced and certified, wherever possible, 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 12mm. The rear portion of the disc, to a depth of ~3mm, 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 this certificate. This certification will therefore expire in September 2031, 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.