

CERTIFICATE OF ANALYSIS

71X PB4 (batch A)

Certified Reference Material Information

Type: TIN WITH IMPURITIES (CAST)
Form and Size: Disc 40mm diameter x 15mm thick
Manufactured by: Universal Scientific Laboratory
Certified and Supplied by: MBH Analytical Limited

Assigned Values

Percentage element by weight

Element	Pb	Cu	Bi	Ag	Cd	Zn	Sb	As
Value ¹	4.00	0.0014	(0.002)	0.0047	0.0027	0.0063	0.0059	0.0045
Uncertainty ²	0.05	0.0002	-	0.0002	0.0002	0.0008	0.0005	0.0005

Note: information in parentheses is not certified - it is 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:

MBH ANALYTICAL LIMITED _____

on 5th July 2007

C Eveleigh

Method of Preparation

This reference material was produced from commercial-purity tin, with the traces added as pure elements, binaries or master alloys. The metal was cast from the bulk melt by sequential transfer of aliquots into individual iron chill moulds. At least 1mm was machined from the lower surface of each disc, to minimise 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. One disc was also checked for vertical uniformity.

Using the combined data for 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).

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

Most of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to a national authority. It is part of the 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 primary reference materials.

Usage

Intended use: With optical emission and X-ray fluorescence spectrometers.

Recommended method of use: Tin and its alloys are 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 optimise precision and accuracy. 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.

Chemical Analysis

Analysis was carried out on millings taken from samples representative of the product. It was performed by a panel of laboratories mostly operating within the terms of EN ISO/IEC 17025 - 2000, using documented standard reference methods and validated by appropriate reference materials.

The individual values listed below are the average of each analyst's results.

Analytical Data

Sample	<u>Percentage element by weight</u>							
	Pb	Cu	Bi	Ag	Cd	Zn	Sb	As
1	3.89	0.0009	0.0008	0.0044	0.0024	0.0050	0.0048	0.0039
2	3.933	0.0011	0.0015	0.0044	0.0024	0.0053	0.0054	0.0040
3	3.945	0.0012	0.0016	0.0046	0.0025	0.0054	0.0055	0.0042
4	3.965	0.0014	0.0020	0.0047	0.0025	0.0055	0.0056	0.0042
5	3.98	0.0015	0.0020	0.0047	0.0026	0.0065	0.0056	0.0044
6	3.984	0.0015	0.0021	0.0048	0.0027	0.0066	0.0059	0.0048
7	3.993	0.0016	0.0023	0.0048	0.0028	0.0066	0.0060	0.0049
8	4.03	0.0016	0.0025	0.0048	0.0028	0.0066	0.0062	0.0050
9	4.072	0.0016		0.0050	0.0028	0.0067	0.0064	0.0050
10	4.083	0.0020			0.0028	0.0069	0.0064	0.0050
11	4.11				0.0031	0.0070	0.0069	
12					0.0032	0.0080		
Mean	3.999	0.0014	(0.002)	0.0047	0.0027	0.0063	0.0059	0.0045
Std Dev	0.068	0.0003	-	0.0002	0.0003	0.0009	0.0006	0.0004
C_(95%)	0.046	0.0002	-	0.0002	0.0002	0.0006	0.0004	0.0003

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

Sheffield Assay Office	Sheffield, England	UKAS accreditation 0014
Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Westmoreland Testing and Research, Inc	Youngstown, PA, USA	A2LA accreditation 0621
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Pvt Ltd	Mumbai, India	NABL accreditation 0367
Laboratory TUV Nord-Czech	Brno, Czech Republic	CAI accreditation 1060
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Coleshill Laboratories Ltd	Coleshill, England	
Genitest Inc	Montreal, Canada	
Cookson Electronics, Fry Technologies	Altoona, PA, USA	

Note: to achieve National Accreditation (eg UKAS, NATA, A2LA, CNAL, NABL, CAI), 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
Lead	1, 2, 4-7, 10, 11	-	3, 8, 9	
Copper	1, 3-8	10	2, 9	
Bismuth	1, 2, 5-8	-	4	3 photometric (iodide)
Silver	2, 3, 6-9	5	1, 4	
Cadmium	2-5, 7-9, 11, 12	1	6, 10	
Zinc	1-5, 10-12	8	6, 7, 9	
Antimony	1-5, 7-11	6	-	
Arsenic	1, 3-9	2	-	10 photometric (molybdenum blue)

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, 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. Material to the rear of the disc, to a depth of ~5mm, 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 July 2027, although we reserve the right to make changes as issue revisions, in the intervening period.

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.