

# CERTIFICATE OF ANALYSIS

**31X 7835.11 (batch A)**

## Certified Reference Material Information

Type: LEADED BRASS (CHILL CAST)  
Form and Size: Disc ~40mm diameter  
Manufactured by: Polycast Ltd  
Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	Sn	Pb	Fe	Ni	Al	Cu	Mn	Sb
Value <sup>1</sup>	<b>0.1522</b>	<b>1.695</b>	<b>0.192</b>	<b>0.1007</b>	<b>0.908</b>	<b>65.75</b>	<b>0.0101</b>	<b>0.0122</b>
Uncertainty <sup>2</sup>	0.0015	0.012	0.005	0.0012	0.009	0.08	0.0004	0.0006

Element	Co	As	P	Si	Bi	Te	Zn
Value <sup>1</sup>	<b>0.0065</b>	<b>0.143</b>	<b>0.024</b>	<b>0.010</b>	<b>0.011</b>	<b>(0.009)</b>	<b>30.98</b>
Uncertainty <sup>2</sup>	0.0003	0.004	0.001	0.001	0.001	-	0.09

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:

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

on 25<sup>th</sup> May 2016

C. Eveleigh

## **Method of Preparation**

This reference material was produced from commercial-purity metals, and master alloys. The discs are the product of one melt poured into a sequence of multiple chill moulds with feeding systems designed to ensure sound discs. At least 2mm has been removed from the chill-cast faces of the discs to minimise 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 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 - 2005, using documented standard reference methods and validated by appropriate reference materials.

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, described 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: Copper alloys are generally prepared by machining on a mill or 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.

For optical emission spectroscopy, 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	Sn	Pb	Fe	Ni	Al	Cu	Mn	Sb
1	0.1490	1.680	0.182	0.0989	0.900	65.63	0.0092	0.0103
2	0.1496	1.687	0.182	0.0996	0.900	65.67	0.0094	0.0109
3	0.1499	1.690	0.182	0.1002	0.901	65.68	0.0095	0.0109
4	0.1502	1.695	0.189	0.1005	0.901	65.70	0.0098	0.0109
5	0.1519	1.695	0.190	0.1008	0.902	65.72	0.0098	0.0114
6	0.1520	1.697	0.193	0.1009	0.905	65.74	0.0099	0.0115
7	0.1520	1.699	0.193	0.1010	0.912	65.75	0.0100	0.0119
8	0.1522	1.700	0.196	0.1010	0.924	65.75	0.0101	0.0123
9	0.1530	1.702	0.196	0.1012	0.924	65.85	0.0101	0.0125
10	0.1530	1.709	0.197	0.1020		65.88	0.0102	0.0126
11	0.1540		0.199	0.1021		65.90	0.0104	0.0126
12	0.1540		0.200				0.0105	0.0129
13	0.1549						0.0106	0.0131
14	0.1550						0.0108	0.0136
15							0.0109	0.0136
16							0.0110	0.0136
<b>Mean</b>	<b>0.1522</b>	<b>1.695</b>	<b>0.192</b>	<b>0.1007</b>	<b>0.908</b>	<b>65.75</b>	<b>0.0101</b>	<b>0.0122</b>
<b>Std Dev</b>	0.0019	0.008	0.007	0.0009	0.010	0.09	0.0005	0.0011
<b>C<sub>(95%)</sub></b>	0.0011	0.006	0.004	0.0006	0.008	0.06	0.0003	0.0006

Sample	Co	As	P	Si	Bi	Te	Zn
1	0.0060	0.134	0.0216	0.0072	0.0091	0.0067	30.79
2	0.0062	0.135	0.0229	0.0076	0.0094	0.0069	30.82
3	0.0063	0.135	0.0233	0.0078	0.0097	0.0086	30.90
4	0.0063	0.139	0.0238	0.0078	0.0098	0.0098	30.96
5	0.0063	0.142	0.0239	0.0094	0.0102	0.0103	30.98
6	0.0063	0.142	0.0240	0.0098	0.0113		31.00
7	0.0065	0.142	0.0245	0.0104	0.0114		31.01
8	0.0067	0.144	0.0245	0.0107	0.0116		31.02
9	0.0069	0.147	0.0255	0.0107	0.0117		31.05
10	0.0070	0.149		0.0121	0.0121		31.08
11	0.0071	0.151		0.0125	0.0123		31.11
12		0.151		0.0135	0.0124		
13		0.151					
<b>Mean</b>	<b>0.0065</b>	<b>0.143</b>	<b>0.0238</b>	<b>0.0100</b>	<b>0.0109</b>	<b>0.0085</b>	<b>30.98</b>
<b>Std Dev</b>	0.0004	0.006	0.0011	0.0021	0.0012	0.0016	0.10
<b>C<sub>(95%)</sub></b>	0.0003	0.004	0.0008	0.0013	0.0008	0.0020	0.07

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, NSW, Australia	NATA accreditation	0492
Genitest Inc.	Montreal, QC, Canada	PRI accreditation	123077
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation	0173
Shanghai JinYi Test Technology Co. Ltd	Shanghai, China	CNAL accreditation	0783
Shandong Metallurgical & Science Research Bureau Veritas CPS Ltd.	Jinan, Shandong, China	CNAS accreditation	1461
TCR Engineering Services pvt Ltd.	Chennai, India	NABL accreditation	0025
Raghavendra Spectro Metallurgical Laboratory	Mumbai, India	NABL accreditation	0367
Institute of Non-ferrous Metals	Bangalore, India	NABL accreditation	0371
TEC Eurolab SRL	Gliwice, Poland	PCA accreditation	AB274
AMG Superalloys UK Ltd	Modena, Italy	ACCREDIA accreditation	52
Analyticka Laborator Lithea sro	Rotherham, UK		
Colonial Metal Co.	Brno, Czech Republic		
Coleshill Laboratories Limited	Columbia, PA, USA		
	Coleshill, UK		

Note: to achieve the above accreditation (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	XRF	FAAS		OTHER
Tin	2-7, 10, 12-14	11	1, 8	9	photometric (phenyl fluorone)
Lead	1, 2, 4, 5, 7, 9, 10	-	3, 6, 8		
Iron	1-3, 5-10, 12	-	11	4	photometric (orthophenanthroline)
Nickel	1, 3-6, 8, 10, 11	-	2, 9	7	photometric (Dimethyl glyoxime)
Aluminium	2-9	-	1		
Copper	9, 11	-	-	1, 4-6, 10 2, 3, 7, 8	volumetric (thiosulfate) electrogravimetric
Manganese	1, 2, 4, 5, 7-10, 12-16	-	3, 6, 11		
Antimony	1-4, 8, 10-16	-	5, 6, 9	7	photometric (crystal violet)
Cobalt	1-4, 6, 8, 9, 11	-	5, 7, 10		
Arsenic	1, 3, 6-13	-	4, 5	2	photometric (turbidity)
Phosphorus	1, 3-7	-	-	8 2, 9	photometric (molybdenum blue) volumetric (alkalimetric)
Silicon	2, 4-6, 11, 12	-	-	1, 8-10 3, 7	photometric (molybdenum blue) gravimetric (perchloric acid)
Bismuth	1, 3-7, 9, 10, 12	-	2, 8	11	photometric (iodide)
Tellurium	1-5	-	-		
Zinc	5, 6, 11	3	-	1, 2, 4, 7-10	volumetric (EDTA)

## 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. Material to the rear of the disc, to a depth of ~3 mm, is not certified.

This material will 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. Technical support for this certification will therefore expire in May 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.