

# CERTIFICATE OF ANALYSIS

## 32X LB15 (batch E)

### Certified Reference Material Information

Type: LEADED BRONZE (CHILL CAST)  
Form and Size: Disc 40mm diameter x ~15mm thickness  
Manufactured by: Polycast Ltd  
Certified and Supplied by: MBH Analytical Ltd

### Assigned Values

#### Percentage element by weight

Element	Sn	Pb	Zn	Fe	Ni	Co	Si	As
Value <sup>1</sup>	4.51	21.42	0.064	0.0012	0.123	0.0051	0.0008	0.0273
Uncertainty <sup>2</sup>	0.05	0.14	0.002	0.0003	0.002	0.0002	0.0002	0.0009

Element	Bi	Sb	Al	P	Ag	S	Cu
Value <sup>1</sup>	(0.13)	0.127	(0.001)	0.0035	0.0217	0.032	73.60
Uncertainty <sup>2</sup>	-	0.002	-	0.0004	0.0006	0.004	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 \_\_\_\_\_

C Eveleigh

on 10th January 2014



## **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. Approximately 2mm has been removed from the cast faces of the discs to minimise surface effects.

## **Sampling**

Samples for chemical analysis were taken from various positions throughout the casting process. At least 15% 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 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, 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: 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.

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	Zn	Fe	Ni	Co	Si	As
1	4.390	21.19	0.0596	0.0004	0.118	0.0047	0.0003	0.0244
2	4.432	21.26	0.0600	0.0006	0.118	0.0047	0.0007	0.0253
3	4.465	21.28	0.0615	0.0009	0.119	0.0049	0.0008	0.0263
4	4.470	21.33	0.0618	0.0010	0.120	0.0049	0.0008	0.0263
5	4.471	21.36	0.0620	0.0013	0.122	0.0050	0.0008	0.0265
6	4.490	21.36	0.0624	0.0014	0.122	0.0051	0.0008	0.0265
7	4.512	21.38	0.0635	0.0014	0.123	0.0051	0.0009	0.0270
8	4.518	21.41	0.0642	0.0015	0.123	0.0051	0.0010	0.0272
9	4.529	21.46	0.0652	0.0015	0.124	0.0053	0.0010	0.0273
10	4.530	21.49	0.0655	0.0017	0.125	0.0053		0.0282
11	4.563	21.65	0.0657	0.0018	0.125	0.0053		0.0282
12	4.588	21.66	0.0665		0.126	0.0054		0.0283
13	4.598	21.67	0.0677		0.126	0.0054		0.0284
14	4.624		0.0683		0.128	0.0055		0.0293
15			0.0683		0.129			0.0304
<b>Mean</b>	<b>4.513</b>	<b>21.42</b>	<b>0.0641</b>	<b>0.0012</b>	<b>0.123</b>	<b>0.0051</b>	<b>0.0008</b>	<b>0.0273</b>
<b>Std Dev</b>	0.066	0.16	0.0029	0.0004	0.003	0.0003	0.0002	0.0015
<b>C<sub>(95%)</sub></b>	0.038	0.09	0.0016	0.0003	0.002	0.0001	0.0002	0.0009

Sample	Bi	Sb	Al	P	Ag	S	Cu
1	0.119	0.123	0.0002	0.0028	0.0200	0.0258	73.50
2	0.124	0.124	0.0004	0.0028	0.0206	0.0290	73.51
3	0.124	0.124	0.0005	0.0029	0.0211	0.0294	73.60
4	0.125	0.125	0.0007	0.0033	0.0211	0.0313	73.61
5	0.126	0.126	0.0009	0.0034	0.0212	0.0321	73.65
6	0.128	0.126	0.0010	0.0034	0.0212	0.0334	73.67
7	0.129	0.127	0.0011	0.0035	0.0214	0.0385	73.69
8	0.131	0.128	0.0012	0.0036	0.0215	0.0388	
9	0.131	0.128	0.0014	0.0040	0.0217		
10	0.139	0.129	0.0014	0.0042	0.0223		
11		0.129	0.0018	0.0043	0.0224		
12		0.130	0.0019		0.0228		
13		0.130	0.0022		0.0229		
14		0.130	0.0023		0.0235		
<b>Mean</b>	<b>0.128</b>	<b>0.127</b>	<b>(0.0012)</b>	<b>0.0035</b>	<b>0.0217</b>	<b>0.032</b>	<b>73.60</b>
<b>Std Dev</b>	0.005	0.002	-	0.0005	0.0010	0.005	0.07
<b>C<sub>(95%)</sub></b>	0.004	0.001	-	0.0004	0.0006	0.004	0.07

Note: C<sub>(95%)</sub> 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, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Bureau Veritas Pvt Ltd	Chennai, India	NABL accreditation 0025
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Mineral & Metallurgical Laboratories	Bangalore, India	
Colonial Metals Co	Columbia, PA, USA	
London & Scandinavian Met. Co	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Lithea sro	Brno, Czech Republic	

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, 3, 5-7, 9, 12, 13	1	8	4, 13, 14 10 11 volumetric (iodate) photometric (phenyl fluorone) gravimetric
Lead	5-10, 12	-	1	2, 4, 13 3 11 electrogravimetric volumetric (EDTA) gravimetric (sulfate)
Zinc	1, 3-5, 710, 13, 14	2	6, 11, 12, 15	
Iron	2, 5-9, 11	-	1, 3, 10	4 photometric (orthophenanthroline)
Nickel	1-4, 6, 7, 11, 13, 14	15	8-10, 12	5 gravimetric (dimethyl glyoxime)
Cobalt	1-3, 5, 7-11, 13, 14	-	4, 6, 12	
Silicon	1, 3-5	-	-	2, 6-9 photometric (molybdenum blue)
Arsenic	1, 3-6, 9-11, 13-15	7	8, 12	2 photometric (turbidity)
Bismuth	1, 5-10	-	2-4	
Antimony	1-3, 5, 6, 8-10, 13	12	7, 11, 14	4 photometric (crystal violet)
Aluminium	1, 2, 4, 6-8, 10-13	-	3, 5, 9	14 photometric (chrome azurol S)
Phosphorus	3, 6-9, 11	-	-	1, 2 volumetric (alkalimetric)
				4, 5, 10 photometric (molybdenum yellow)
Silver	1, 3, 5-7, 9-13	-	2, 4, 8, 14	
Sulfur	5	2	-	
Copper	4	-	-	1, 3, 5 2, 6, 7 electrogravimetric volumetric (thiosulfate)

## Notes

This Certified Reference Material has been produced and certified 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. Material to the rear of the disc, to a depth of ~3mm, is not certified.

This material 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 January 2034, 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.