

# CERTIFICATE OF ANALYSIS

**31X 7835.9 (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	S
Value <sup>1</sup>	1.48	1.024	0.408	0.100	0.092	78.48	0.0009	0.445	0.0161
Uncertainty <sup>2</sup>	0.02	0.017	0.003	0.002	0.003	0.11	0.0001	0.003	0.0010

Element	Co	As	P	Si	Bi	Se	Cd	Ag	Zn
Value <sup>1</sup>	0.0813	0.107	0.0390	0.0232	0.81	0.34	0.0673	2.12	14.34
Uncertainty <sup>2</sup>	0.0013	0.002	0.0006	0.0014	0.02	0.02	0.0015	0.04	0.06

## 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 14<sup>th</sup> July 2012

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**

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, 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.

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	S
1	1.445	0.989	0.402	0.0947	0.0885	78.31	0.0006	0.438	0.0140
2	1.450	0.992	0.403	0.0950	0.0891	78.38	0.0008	0.439	0.0149
3	1.451	0.995	0.407	0.0966	0.0894	78.38	0.0009	0.440	0.0152
4	1.459	1.019	0.409	0.0967	0.0894	78.43	0.0009	0.442	0.0155
5	1.471	1.022	0.410	0.0990	0.0900	78.48	0.0009	0.444	0.0162
6	1.472	1.030	0.410	0.0990	0.0901	78.62	0.0009	0.446	0.0164
7	1.484	1.032	0.410	0.0996	0.0911	78.62	0.0009	0.446	0.0168
8	1.494	1.033	0.410	0.1024	0.0912	78.64	0.0010	0.447	0.0178
9	1.497	1.040	0.411	0.1029	0.0918		0.0011	0.447	0.0183
10	1.520	1.042	0.412	0.1030	0.0945			0.449	
11	1.522	1.045		0.1034	0.0956			0.450	
12	1.527	1.054		0.1045	0.0960			0.450	
13				0.1048	0.0966				
14					0.0967				
<b>Mean</b>	<b>1.483</b>	<b>1.024</b>	<b>0.408</b>	<b>0.1001</b>	<b>0.0921</b>	<b>78.48</b>	<b>0.0009</b>	<b>0.445</b>	<b>0.0161</b>
<b>Std Dev</b>	0.029	0.022	0.003	0.0036	0.0031	0.13	0.0001	0.004	0.0014
<b>C<sub>(95%)</sub></b>	0.019	0.014	0.002	0.0022	0.0018	0.11	0.0001	0.003	0.0010

Sample	Co	As	P	Si	Bi	Se	Cd	Ag	Zn
1	0.0777	0.103	0.0370	0.0193	0.805	0.325	0.0644	2.034	14.25
2	0.0787	0.103	0.0379	0.0214	0.809	0.327	0.0656	2.080	14.26
3	0.0794	0.104	0.0382	0.0220	0.809	0.327	0.0657	2.092	14.29
4	0.0796	0.105	0.0385	0.0230	0.813	0.337	0.0662	2.099	14.30
5	0.0805	0.106	0.0385	0.0234	0.814	0.344	0.0666	2.099	14.31
6	0.0806	0.107	0.0389	0.0236	0.815	0.344	0.0669	2.102	14.32
7	0.0810	0.109	0.0390	0.0238	0.820	0.366	0.0676	2.103	14.35
8	0.0811	0.109	0.0391	0.0239	0.827		0.0685	2.138	14.39
9	0.0817	0.109	0.0394	0.0253			0.0690	2.160	14.41
10	0.0824	0.110	0.0398	0.0262			0.0698	2.161	14.43
11	0.0827	0.111	0.0398				0.0701	2.218	14.45
12	0.0833	0.111	0.0400						
13	0.0845		0.0402						
14	0.0856		0.0402						
<b>Mean</b>	<b>0.0813</b>	<b>0.107</b>	<b>0.0390</b>	<b>0.0232</b>	<b>0.814</b>	<b>0.339</b>	<b>0.0673</b>	<b>2.117</b>	<b>14.34</b>
<b>Std Dev</b>	0.0022	0.003	0.0009	0.0020	0.007	0.015	0.0019	0.049	0.07
<b>C<sub>(95%)</sub></b>	0.0013	0.002	0.0005	0.0014	0.006	0.013	0.0012	0.033	0.05

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, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Colonial Metals Co	Columbia, PA, USA	
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Coleshill Laboratories Ltd	Birmingham, England	
London & Scandinavian Met Co	Rotherham, England	
LECO Corporation	St Joseph, MI, USA	

Note: to achieve the above 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	GD-AES	OTHER	
Tin	1-4, 10-12	5, 7	-	6, 8, 9	photometric (phenyl fluorone) volumetric (iodate)
Lead	1-6, 11, 12	8-10	-	7	electrogravimetric
Iron	2-4, 7-9	1, 5, 6	10		
Nickel	1, 3, 4, 8-10, 12, 13	5, 7, 11	2	6	photometric (dimethyl glyoxime)
Aluminium	1, 2, 5, 6, 8, 9, 11, 14	3, 4, 7	12	10, 13	photometric (chrome azurol S)
Copper	2, 3	-	-	1, 5	electrogravimetric
				4	XRF
				6-8	volumetric (thiosulfate)
Manganese	1, 4-7, 9	2, 3	-	8	ICP-MS
Antimony	1, 2, 4, 6, 8-10	3, 5, 7, 11	-		
Sulfur	6, 8	-	-	1	XRF
				2-5, 7, 9	combustion (infra-red detection)
Cobalt	1, 2, 4, 5, 9-11, 13, 14	3, 6, 8, 12	7		
Arsenic	1-8	9-11	-		
Phosphorus	2, 4-6, 8, 9, 11-13	-	7	1, 10, 14	photometric (molybdenum yellow)
				3	volumetric (alkalimetric)
Silicon	2, 4, 5, 9	-	3	1, 6-8, 10	photometric (molybdenum blue)
Bismuth	2-5, 7, 8	6	1		
Selenium	2-7	1	-		
Cadmium	1,3, 4, 7, 8, 10	2, 5, 6, 11	9		
Silver	2, 5, 6, 11	1, 3, 7-9	10	4	volumetric (thiocyanate)
Zinc	1, 4, 10		3	2	XRF
				5-9, 11	volumetric (EDTA)

## Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 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 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 2032, 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.