

# CERTIFICATE OF ANALYSIS

**32X PB14 (batch E)**

## Certified Reference Material Information

Type: PHOSPHOR BRONZE (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	Zn	Fe	Ni	Mn	Al	Si
Value <sup>1</sup>	<b>9.65</b>	<b>0.0354</b>	<b>0.103</b>	<b>0.0211</b>	<b>0.103</b>	<b>0.0141</b>	<b>0.0201</b>	<b>(0.003)</b>
Uncertainty <sup>2</sup>	0.04	0.0014	0.003	0.0009	0.002	0.0004	0.0009	-

Element	As	P	Co	Sb	Bi	Ag	S	Cu
Value <sup>1</sup>	<b>0.0235</b>	<b>0.128</b>	<b>0.0047</b>	<b>0.0433</b>	<b>0.146</b>	<b>0.0152</b>	<b>0.070</b>	<b>89.70</b>
Uncertainty <sup>2</sup>	0.0009	0.003	0.0002	0.0015	0.003	0.0004	0.003	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 23<sup>rd</sup> July 2017

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. 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**

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. The testing procedure was in accordance with ASTM E826 and the material found acceptable.

From this test data, through-batch variation values were derived for each element as an indicator of any minor compositional variation (as determined for the specific sample size and other limitations of 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, 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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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	Mn	Al	Si
1	9.592	0.0327	0.0982	0.0199	0.0983	0.0131	0.0180	0.0021
2	9.606	0.0330	0.0991	0.0201	0.0998	0.0135	0.0193	0.0021
3	9.607	0.0333	0.1010	0.0202	0.1018	0.0138	0.0194	0.0026
4	9.609	0.0345	0.1016	0.0203	0.1020	0.0138	0.0195	0.0030
5	9.620	0.0356	0.1021	0.0204	0.1028	0.0139	0.0195	0.0032
6	9.624	0.0357	0.1030	0.0205	0.1033	0.0139	0.0197	0.0033
7	9.660	0.0358	0.1030	0.0205	0.1050	0.0140	0.0198	0.0040
8	9.660	0.0364	0.1049	0.0209	0.1057	0.0141	0.0204	0.0046
9	9.677	0.0366	0.1060	0.0212	0.1068	0.0142	0.0205	
10	9.686	0.0367	0.1080	0.0218	0.1070	0.0143	0.0205	
11	9.710	0.0367		0.0219		0.0147	0.0208	
12	9.718	0.0377		0.0221		0.0147	0.0208	
13				0.0225		0.0147	0.0210	
14				0.0235		0.0148	0.0222	
<b>Mean</b>	<b>9.647</b>	<b>0.0354</b>	<b>0.1027</b>	<b>0.0211</b>	<b>0.1033</b>	<b>0.0141</b>	<b>0.0201</b>	<b>0.0031</b>
<b>Std Dev</b>	0.043	0.0016	0.0030	0.0011	0.0029	0.0005	0.0010	0.0009
<b>C<sub>(95%)</sub></b>	0.028	0.0010	0.0022	0.0006	0.0021	0.0003	0.0006	0.0007

Sample	As	P	Co	Sb	Bi	Ag	S	Cu
1	0.0211	0.1216	0.0041	0.0394	0.1396	0.0139	0.0660	89.51
2	0.0218	0.1219	0.0041	0.0400	0.1410	0.0145	0.0661	89.59
3	0.0218	0.1231	0.0043	0.0408	0.1410	0.0148	0.0677	89.60
4	0.0221	0.1239	0.0044	0.0416	0.1440	0.0149	0.0692	89.63
5	0.0228	0.1250	0.0045	0.0420	0.1444	0.0149	0.0698	89.65
6	0.0236	0.1260	0.0046	0.0425	0.1450	0.0149	0.0699	89.72
7	0.0239	0.1270	0.0047	0.0433	0.1452	0.0149	0.0703	89.79
8	0.0240	0.1281	0.0047	0.0435	0.1459	0.0151	0.0735	89.82
9	0.0242	0.1290	0.0047	0.0440	0.1463	0.0153	0.0748	89.86
10	0.0245	0.1295	0.0048	0.0440	0.1480	0.0153	0.0756	89.87
11	0.0246	0.1315	0.0048	0.0443	0.1480	0.0157		
12	0.0249	0.1330	0.0049	0.0446	0.1495	0.0157		
13	0.0258	0.1340	0.0051	0.0450	0.1505	0.0159		
14		0.1369	0.0051	0.0468	0.1513	0.0163		
15			0.0057	0.0472				
<b>Mean</b>	<b>0.0235</b>	<b>0.1279</b>	<b>0.0047</b>	<b>0.0433</b>	<b>0.1457</b>	<b>0.0152</b>	<b>0.0703</b>	<b>89.70</b>
<b>Std Dev</b>	0.0014	0.0047	0.0004	0.0023	0.0036	0.0006	0.0034	0.13
<b>C<sub>(95%)</sub></b>	0.0009	0.0027	0.0002	0.0013	0.0021	0.0004	0.0024	0.09

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  
Sheffield Assay Office  
Universal Scientific Laboratory Pty Ltd  
Shanghai Jinyi Test Technology Co  
Luo Yang Copper  
Shandong Metallurgical & Science Research  
Genitest, Inc  
Raghavendra Spectromet Laboratory  
TCR Engineering Services Pvt Ltd  
Institute of Non-Ferrous Metals  
TEC-Eurolab SRL  
INCDMNR-IMNR  
Mineral & Metallurgical Laboratories  
AMG Superalloys UK Ltd  
Coleshill Laboratories Ltd  
Analyticka Laborator Lithea sro

Middlesbrough, England  
Sheffield, England  
Milperra, NSW, Australia  
Shanghai, China  
Luo Yang, He Nan, China  
Jinan, Shandong, China  
Montreal, Canada  
Bangalore, India  
Mumbai, India  
Gliwice, Poland  
Modena, Italy  
Pantelimon, Romania  
Bangalore, India  
Rotherham, England  
Birmingham, England  
Brno, Czech Republic

UKAS accreditation 0239  
UKAS accreditation 0012  
NATA accreditation 0492  
CNAL accreditation 0783  
CNAL accreditation 0173  
CNAS accreditation 1461  
PRI accreditation 123077  
NABL accreditation 0371  
NABL Accreditation 0367  
PCA accreditation AB274  
Accredia accreditation 52

Note: to achieve the above accreditation (UKAS, 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	
Tin	4, 8, 9, 11, 12	2	1 3, 6, 7, 10 5 3 3 6 13 2, 6 7 14 3, 5, 6, 8 10 2, 12, 14 6, 7	gravimetric (metastannic acid) volumetric (iodate) photometric (phenyl fluorone) gravimetric (sulfate) volumetric (EDTA) volumetric (redox) photometric (orthophenthroline) photometric (dimethyl glyoxime) photometric (chrome azurol S) volumetric (EDTA) photometric (molybdenum blue) photometric (turbidity) volumetric (alkalimetric) photometric (molybdenum yellow)
Lead	1, 2, 4-6, 8, 9, 11, 12	7, 10		
Zinc	1, 2, 4, 7-10	5, 6		
Iron	1-5, 7, 8, 10, 11, 14	9, 12		
Nickel	1, 3, 5, 7-9	4, 10		
Manganese	1-3, 5, 6, 8, 10-14	4, 7, 9		
Aluminium	1-3, 5, 6, 8-13	4		
Silicon	1, 2, 4, 7	-		
Arsenic	1-9, 11, 13	12		
Phosphorus	1, 3-5, 8-11, 13	-		
Cobalt	2-9, 11, 13, 15	1, 10, 14		
Antimony	1-6, 9, 10, 12-14	11, 15		
Bismuth	1-3, 5, 6, 8, 10-12, 14	7, 9		
Silver	2-8, 12-14	9-11		
Sulfur	3, 7	-		
Copper		-		

## Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, 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 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 July 2037, 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.