

# CERTIFICATE OF ANALYSIS

**31X TB1 (batch K)**

## Certified Reference Material Information

Type: TRACE ELEMENTS IN 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	Zn	Fe	Mn	Ni	Si	As	Co
Value <sup>1</sup>	0.134	0.207	35.17	0.171	0.280	0.0619	0.080	0.111	0.0500
Uncertainty <sup>2</sup>	0.003	0.004	0.10	0.003	0.003	0.0012	0.003	0.002	0.0011

Element	P	Al	Bi	Sb	Ag	B	Cd	Cu
Value <sup>1</sup>	0.0201	0.493	0.0414	0.105	0.0455	0.0010	0.0118	63.08
Uncertainty <sup>2</sup>	0.0005	0.007	0.0013	0.002	0.0008	0.0001	0.0008	0.09

## 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 4<sup>th</sup> November 2016

C. Eveleigh

## **Method of Preparation**

This reference material was produced from commercial-purity metals, binaries 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 - 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 compositional variation 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: Coppers 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-elemental effects.

## Analytical Data

### Percentage element by weight

Sample	Sn	Pb	Zn	Fe	Mn	Ni	Si	As	Co
1	0.1271	0.1968	35.050	0.1655	0.2740	0.0592	0.0739	0.1079	0.0477
2	0.1280	0.1974	35.080	0.1677	0.2747	0.0598	0.0745	0.1083	0.0490
3	0.1291	0.1998	35.110	0.1681	0.2760	0.0599	0.0783	0.1090	0.0494
4	0.1294	0.2033	35.157	0.1688	0.2768	0.0603	0.0789	0.1090	0.0498
5	0.1300	0.2059	35.186	0.1692	0.2773	0.0605	0.0791	0.1095	0.0500
6	0.1322	0.2060	35.196	0.1703	0.2798	0.0611	0.0793	0.1100	0.0502
7	0.1323	0.2066	35.201	0.1705	0.2799	0.0611	0.0799	0.1104	0.0503
8	0.1346	0.2090	35.210	0.1710	0.2801	0.0618	0.0801	0.1104	0.0503
9	0.1348	0.2097	35.222	0.1710	0.2802	0.0623	0.0822	0.1115	0.0508
10	0.1357	0.2099	35.262	0.1740	0.2820	0.0624	0.0834	0.1120	0.0511
11	0.1368	0.2105		0.1750	0.2820	0.0630	0.0840	0.1140	0.0514
12	0.1370	0.2110		0.1750	0.2824	0.0631	0.0849	0.1150	
13	0.1370	0.2110			0.2850	0.0632	0.0867	0.1153	
14	0.1405	0.2114			0.2850	0.0634		0.1160	
15	0.1420	0.2123			0.2880	0.0641			
16						0.0650			
<b>Mean</b>	<b>0.1338</b>	<b>0.2067</b>	<b>35.167</b>	<b>0.1705</b>	<b>0.2802</b>	<b>0.0619</b>	<b>0.0804</b>	<b>0.1113</b>	<b>0.0500</b>
<b>Std Dev</b>	0.0045	0.0052	0.067	0.0030	0.0040	0.0017	0.0038	0.0027	0.0010
<b>C<sub>(95%)</sub></b>	0.0025	0.0029	0.048	0.0019	0.0022	0.0009	0.0023	0.0016	0.0007

Sample	P	Al	Bi	Sb	Ag	B	Cd	Cu
1	0.0189	0.4700	0.0393	0.0997	0.0437	0.0009	0.0113	62.990
2	0.0193	0.4733	0.0394	0.0998	0.0443	0.0009	0.0113	62.994
3	0.0195	0.4756	0.0399	0.1020	0.0451	0.0009	0.0116	63.019
4	0.0198	0.4831	0.0399	0.1023	0.0451	0.0009	0.0117	63.070
5	0.0200	0.4896	0.0400	0.1030	0.0452	0.0009	0.0117	63.081
6	0.0202	0.4902	0.0404	0.1030	0.0453	0.0010	0.0118	63.100
7	0.0203	0.4910	0.0407	0.1050	0.0460	0.0011	0.0119	63.110
8	0.0205	0.4917	0.0409	0.1054	0.0461	0.0012	0.0120	63.133
9	0.0205	0.4977	0.0413	0.1060	0.0464	0.0012	0.0120	63.150
10	0.0209	0.4985	0.0414	0.1060	0.0466	0.0012	0.0123	63.163
11	0.0214	0.4997	0.0425	0.1065	0.0469		0.0124	
12		0.5060	0.0435	0.1070				
13		0.5060	0.0435	0.1080				
14		0.5076	0.0443	0.1110				
15		0.5110	0.0447	0.1112				
<b>Mean</b>	<b>0.0201</b>	<b>0.4927</b>	<b>0.0414</b>	<b>0.1051</b>	<b>0.0455</b>	<b>0.0010</b>	<b>0.0118</b>	<b>63.081</b>
<b>Std Dev</b>	0.0007	0.0129	0.0018	0.0035	0.0010	0.0001	0.0004	0.063
<b>C<sub>(95%)</sub></b>	0.0005	0.0071	0.0010	0.0019	0.0007	0.0001	0.0002	0.045

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, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
AnchorCert Analytical	Birmingham, UK	UKAS accreditation	0667
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
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation	1461
Bureau Veritas CPS Ltd.	Chennai, India	NABL accreditation	0025
TCR Engineering Services PVT. Ltd.	Mumbai, India	NABL accreditation	0367
Raghavendra Spectro Metallurgical Laboratory	Bangalore, India	NABL accreditation	0371
Institute of Non-ferrous Metals	Gliwice, Poland	PCA accreditation	AB274
TEC Eurolab SRL	Modena, Italy	ACCREDIA accreditation	52
Mineral and Metallurgical Laboratories	Bangalore, India		
AMG Superalloys UK Ltd	Rotherham, UK		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
Coleshill Laboratories Ltd	Coleshill, UK		

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	1-3, 5-10, 12, 14	11, 13	4 15	photometric (phenyl fluorone) volumetric (iodide)
Lead	1-4, 7, 9, 10, 13-15	5, 6, 8, 11	12	electrogravimetric
Zinc	4, 5, 8	-	1-3, 6, 7, 9, 10	volumetric (EDTA)
Iron	2-4, 6-8, 10-11	1, 5, 12	9	volumetric (Redox)
Manganese	2, 3, 5-9, 11-13	1, 4, 14, 15	10	volumetric (arsenite)
Nickel	1-4, 7, 8, 11-14, 16	6, 9, 15	5	gravimetric (dimethyl glyoxime)
Silicon	3, 4, 6, 8, 10-11, 13	-	10 1, 12	photometric (dimethyl glyoxime) photometric (molybdenum blue)
Arsenic	1-9, 11-14	10	2, 5, 7, 9	gravimetric (perchloric acid)
Cobalt	2-5, 8-11	1, 6	7	gravimetric (oxide)
Phosphorus	2, 6, 9-11	-	1, 3	photometric (molybdenum yellow)
Aluminium	2, 4-6, 8-14	3, 7	4, 5, 7, 8	volumetric (alkalimetric)
Bismuth	1, 3-5, 7-14	2, 6	1	photometric (chrome azurol S)
Antimony	1, 2, 4-9, 11-13, 15	10	15	volumetric (EDTA) photometric (iodide)
Silver	1-5, 7, 10, 11	8, 9	3	photometric (crystal violet)
Boron	2-10	1	14	volumetric (bromate)
Cadmium	1, 2, 5-11	3, 4	6	volumetric (thioacetamide)
Copper	3, 6, 8	-	1, 2, 4, 5 7, 9, 10	volumetric (thiosulfate) electro-gravimetric

## 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 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 November 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