

# CERTIFICATE OF ANALYSIS

**39X 17873 (batch B)**

## Certified Reference Material Information

Type: RESIDUALS IN COPPER (CHILL-CAST)  
Form and Size: Disc ~40mm diameter  
Produced by: Polycast Ltd  
Certified and supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	Pb	Zn	Sn	Fe	Ni	As
Value <sup>1</sup>	0.059	0.0160	0.0247	0.0052	0.0280	0.0197
Uncertainty <sup>2</sup>	0.003	0.0006	0.0016	0.0003	0.0007	0.0007

Element	Sb	Cd	Bi	S	Se	Ag
Value <sup>1</sup>	0.0202	0.0023	0.0254	0.0095	0.0143	0.0291
Uncertainty <sup>2</sup>	0.0010	0.0002	0.0012	0.0006	0.0004	0.0007

Element	Mn	Cr	Co	P	In	Te
Value <sup>1</sup>	0.0005	(0.0003)	0.0004	0.0009	0.0040	0.0144
Uncertainty <sup>2</sup>	0.0001	-	0.0001	0.0002	0.0003	0.0006

- Notes: i) values given in parentheses are not certified - they are provided for information only.  
ii) <sup>1,2</sup>: for definitions, see page 2.

## Certified by:

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

C Eveleigh

on 29<sup>th</sup> February 2016

## **Method of Preparation**

This reference material was produced from commercial-purity copper, and a combination of pure elements and binaries. Discs were produced by sequential casting into iron moulds.

## **Sampling**

Milled samples for chemical analysis, and discs for homogeneity checks, were taken randomly from several positions throughout the casting process. At least 15% of all discs were selected for homogeneity checking.

## **Homogeneity**

The discs were checked for sample and batch uniformity using an optical emission spectrometer. One disc was checked for vertical uniformity using the same method. Using the mean 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.

## **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.

## **Definitions**

- <sup>1</sup> The assigned 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, listed on page 3.
- <sup>2</sup> The analytical uncertainty values are generated from the 95% half-width confidence interval  $C_{(95\%)}$ , which is derived from the wet analysis results, in accordance with the following 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.

## **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 above. 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, also 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.

## **Usage**

Intended use: With optical emission and X-ray fluorescence spectrometers.

Recommended method of use: Copper and its alloys are generally prepared by machining on a mill or 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 optimise precision and accuracy. 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	Pb	Zn	Sn	Fe	Ni	As
1	0.0561	0.0147	0.0213	0.0043	0.0263	0.0178
2	0.0567	0.0149	0.0217	0.0045	0.0264	0.0178
3	0.0568	0.0151	0.0223	0.0047	0.0271	0.0180
4	0.0577	0.0152	0.0230	0.0047	0.0273	0.0181
5	0.0583	0.0152	0.0239	0.0048	0.0274	0.0197
6	0.0584	0.0153	0.0242	0.0049	0.0274	0.0197
7	0.0589	0.0158	0.0247	0.0049	0.0276	0.0197
8	0.0592	0.0161	0.0250	0.0050	0.0280	0.0197
9	0.0597	0.0163	0.0251	0.0053	0.0281	0.0198
10	0.0599	0.0173	0.0253	0.0053	0.0281	0.0202
11	0.0601	0.0173	0.0258	0.0055	0.0286	0.0204
12	0.0609	0.0173	0.0278	0.0057	0.0296	0.0207
13	0.0609	0.0173	0.0280	0.0058	0.0298	0.0208
14	0.0609		0.0282	0.0060	0.0300	0.0209
15				0.0065		0.0220
<b>Mean</b>	<b>0.0589</b>	<b>0.0160</b>	<b>0.0247</b>	<b>0.0052</b>	<b>0.0280</b>	<b>0.0197</b>
<b>Std Dev</b>	0.0016	0.0010	0.0022	0.0006	0.0012	0.0013
<b>C<sub>(95%)</sub></b>	0.0009	0.0006	0.0013	0.0003	0.0007	0.0007

Sample	Sb	Cd	Bi	S	Se	Ag
1	0.0169	0.0020	0.0241	0.0075	0.0129	0.0271
2	0.0183	0.0020	0.0241	0.0081	0.0138	0.0274
3	0.0186	0.0020	0.0250	0.0082	0.0139	0.0276
4	0.0187	0.0021	0.0250	0.0086	0.0141	0.0280
5	0.0198	0.0021	0.0253	0.0090	0.0142	0.0285
6	0.0198	0.0021	0.0253	0.0094	0.0142	0.0286
7	0.0198	0.0022	0.0254	0.0095	0.0144	0.0289
8	0.0199	0.0022	0.0256	0.0095	0.0148	0.0290
9	0.0201	0.0023	0.0260	0.0098	0.0148	0.0295
10	0.0210	0.0024	0.0265	0.0099	0.0149	0.0296
11	0.0215	0.0025	0.0266	0.0101	0.0153	0.0297
12	0.0215	0.0026		0.0101		0.0298
13	0.0223	0.0026		0.0105		0.0306
14	0.0227	0.0026		0.0112		0.0309
15	0.0228	0.0027		0.0113		0.0315
<b>Mean</b>	<b>0.0202</b>	<b>0.0023</b>	<b>0.0254</b>	<b>0.0095</b>	<b>0.0143</b>	<b>0.0291</b>
<b>Std Dev</b>	0.0017	0.0003	0.0008	0.0011	0.0007	0.0013
<b>C<sub>(95%)</sub></b>	0.0010	0.0001	0.0006	0.0006	0.0004	0.0007

Sample	Mn	Cr	Co	P	In	Te
1	0.0003	0.0001	0.0002	0.0005	0.0034	0.0135
2	0.0004	0.0001	0.0002	0.0005	0.0036	0.0138
3	0.0004	0.0002	0.0003	0.0006	0.0036	0.0139
4	0.0004	0.0002	0.0004	0.0009	0.0036	0.0140
5	0.0004	0.0003	0.0004	0.0009	0.0038	0.0140
6	0.0004	0.0003	0.0004	0.0010	0.0038	0.0141
7	0.0004	0.0005	0.0004	0.0010	0.0039	0.0142
8	0.0005	0.0005	0.0004	0.0010	0.0044	0.0142
9	0.0005	0.0006	0.0005	0.0010	0.0044	0.0147
10	0.0005	0.0006	0.0005	0.0011	0.0046	0.0150
11	0.0006		0.0005	0.0012	0.0051	0.0150
12	0.0006		0.0005			0.0151
13	0.0007					0.0152
<b>Mean</b>	<b>0.0005</b>	<b>(0.0003)</b>	<b>0.0004</b>	<b>0.0009</b>	<b>0.0040</b>	<b>0.0144</b>
<b>Std Dev</b>	0.0001	-	0.0001	0.0002	0.0005	0.0006
<b>C<sub>(95%)</sub></b>	0.0001	-	0.0001	0.0002	0.0003	0.0003

For the definition of C<sub>(95%)</sub> see page 2.

## 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
Shanghai Jinyi Test Technology Co	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
Bureau Veritas CPS Pvt	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Microlab	Chennai, India	
Colonial Metals Co	Columbia, PA, USA	
Coleshill Laboratories Ltd	Birmingham, England	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea, sro	Brno, Czech Republic	

Note: to achieve the above accreditation (UKAS, NATA, etc), test houses are required to demonstrate conformity to the general requirements of EN ISO/IEC 17025.

## Analytical Methods Used

ELEMENT	RESULT No. & METHOD		
	ICP-AES	FAAS	OTHER
Lead	2-5, 7, 9-14	1, 6, 8	
Zinc	1, 2, 4-8, 10, 11	3, 9, 12, 13	
Tin	1-4, 6, 8-11, 13	5, 7, 12	14 photometric (phenyl fluorone)
Iron	2-8, 10, 11, 13, 15	1, 12, 14	9 photometric (orthophenanthroline)
Nickel	1-3, 5, 7, 10-14	4, 8, 9	6 photometric (dimethyl glyoxime)
Arsenic	2-4, 6-13, 15	1, 5, 14	
Antimony	1, 3-8, 10-13	2, 9, 15	14 photometric (crystal violet)
Cadmium	3-7, 9-14	1, 2, 8, 15	
Bismuth	1, 2, 6-11	3, 4	5 photometric (iodide)
Sulfur	3, 15	-	1, 2, 4-14 combustion (volumetric or IR detection)
Selenium	1-3, 5-8, 10, 11	4, 9	
Silver	2, 5-9, 11-15	1, 3, 4, 10	
Manganese	1-6, 9-11, 13	8, 12	7 ICP-MS
Chromium	1, 2, 5-9	4, 10	3 ICP-MS
Cobalt	1, 2, 4-6, 9-12	3, 8	7 ICP-MS
Phosphorus	3, 7-10	-	1, 5, 11 volumetric (alkalimetric) 2, 4, 6 photometric (molybdenum yellow)
Indium	3-11	1, 2	
Tellurium	1-13	-	

## 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, to a depth of 12mm; material to the rear, 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 original analysis. Technical support for this certification will therefore expire in February 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.