

# CERTIFICATE OF ANALYSIS

**39X 17868 (batch AG)**

## 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	Fe	Ni	As	Bi	Sb	Co	Ag	P
Value <sup>1</sup>	0.104	0.197	0.110	0.0222	0.0226	0.0308	0.030	0.0248	0.0249	0.051
Uncertainty <sup>2</sup>	0.002	0.005	0.004	0.0008	0.0005	0.0009	0.002	0.0005	0.0008	0.003

Element	Mn	Cd	Au	Al	Sn	Mg	S	Se	Te	In
Value <sup>1</sup>	0.0123	0.0130	0.0101	0.0072	0.103	0.0085	0.022	0.0133	0.0206	0.0076
Uncertainty <sup>2</sup>	0.0005	0.0004	0.0006	0.0011	0.003	0.0010	0.002	0.0010	0.0010	0.0004

## 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:

on 5<sup>th</sup> December 2012

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

C Eveleigh



## **Method of Preparation**

This reference material was produced from commercial-grade copper, 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 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	Pb	Zn	Fe	Ni	As	Bi	Sb	Co	Ag	P
1	0.1000	0.1900	0.1050	0.0200	0.0221	0.0287	0.0272	0.0234	0.0230	0.0479
2	0.1015	0.1904	0.1050	0.0210	0.0221	0.0289	0.0276	0.0236	0.0233	0.0487
3	0.1018	0.1910	0.1056	0.0210	0.0221	0.0299	0.0283	0.0239	0.0236	0.0487
4	0.1020	0.1922	0.1080	0.0210	0.0222	0.0303	0.0285	0.0241	0.0243	0.0489
5	0.1026	0.1963	0.1088	0.0213	0.0223	0.0303	0.0287	0.0246	0.0248	0.0490
6	0.1030	0.1978	0.1093	0.0220	0.0225	0.0307	0.0287	0.0246	0.0248	0.0493
7	0.1037	0.1987	0.1095	0.0221	0.0226	0.0309	0.0296	0.0246	0.0251	0.0497
8	0.1039	0.2000	0.1101	0.0221	0.0227	0.0310	0.0301	0.0247	0.0252	0.0499
9	0.1045	0.2013	0.1123	0.0222	0.0230	0.0311	0.0301	0.0250	0.0252	0.0510
10	0.1049	0.2021	0.1150	0.0224	0.0231	0.0314	0.0302	0.0251	0.0254	0.0535
11	0.1050	0.2022	0.1150	0.0226	0.0233	0.0318	0.0303	0.0254	0.0256	0.0540
12	0.1067	0.2030	0.1164	0.0234	0.0233	0.0323	0.0303	0.0254	0.0265	0.0543
13	0.1080			0.0234	0.0236	0.0329	0.0311	0.0256	0.0270	0.0543
14	0.1082			0.0241			0.0318	0.0256		
15				0.0245				0.0267		
<b>Mean</b>	<b>0.1040</b>	<b>0.1971</b>	<b>0.1100</b>	<b>0.0222</b>	<b>0.0226</b>	<b>0.0308</b>	<b>0.0295</b>	<b>0.0248</b>	<b>0.0249</b>	<b>0.0507</b>
<b>Std Dev</b>	0.0024	0.0050	0.0040	0.0013	0.0005	0.0012	0.0013	0.0009	0.0012	0.0024
<b>C<sub>(95%)</sub></b>	0.0014	0.0032	0.0025	0.0007	0.0003	0.0007	0.0008	0.0005	0.0007	0.0015

Sample	Mn	Cd	Au	Al	Sn	Mg	S	Se	Te	In
1	0.0119	0.0123	0.0088	0.0040	0.0969	0.0067	0.0178	0.0108	0.0195	0.0062
2	0.0120	0.0123	0.0093	0.0041	0.0998	0.0067	0.0194	0.0112	0.0197	0.0063
3	0.0120	0.0124	0.0095	0.0049	0.1005	0.0070	0.0197	0.0113	0.0199	0.0070
4	0.0121	0.0126	0.0096	0.0052	0.1009	0.0072	0.0200	0.0133	0.0202	0.0073
5	0.0121	0.0128	0.0100	0.0059	0.1016	0.0072	0.0207	0.0135	0.0203	0.0076
6	0.0121	0.0129	0.0100	0.0070	0.1022	0.0075	0.0212	0.0138	0.0208	0.0076
7	0.0123	0.0131	0.0108	0.0075	0.1031	0.0082	0.0228	0.0140	0.0208	0.0077
8	0.0125	0.0133	0.0108	0.0075	0.1050	0.0088	0.0229	0.0141	0.0209	0.0078
9	0.0125	0.0133	0.0109	0.0078	0.1056	0.0089	0.0230	0.0145	0.0209	0.0079
10	0.0128	0.0135	0.0111	0.0078	0.1070	0.0091	0.0231	0.0150	0.0211	0.0079
11	0.0130	0.0136		0.0081	0.1086	0.0095	0.0257	0.0152	0.0212	0.0080
12		0.0137		0.0090	0.1100	0.0097	0.0264		0.0215	0.0080
13				0.0098		0.0103				0.0089
14				0.0100		0.0104				
15				0.0101		0.0108				
<b>Mean</b>	<b>0.0123</b>	<b>0.0130</b>	<b>0.0101</b>	<b>0.0072</b>	<b>0.1034</b>	<b>0.0085</b>	<b>0.0219</b>	<b>0.0133</b>	<b>0.0206</b>	<b>0.0076</b>
<b>Std Dev</b>	0.0004	0.0005	0.0008	0.0020	0.0039	0.0014	0.0026	0.0015	0.0006	0.0007
<b>C<sub>(95%)</sub></b>	0.0002	0.0003	0.0006	0.0011	0.0021	0.0008	0.0016	0.0010	0.0004	0.0004

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  
Sheffield Assay Office  
Universal Scientific Laboratory Pty Ltd  
Genitest, Inc  
Institute of Iron & Steel Technology  
Luo Yang Copper  
Sargam Metals Pvt Ltd  
Raghavendra Spectromet Laboratory  
TCR Engineering Services Ltd  
Institute of Non-Ferrous Metals  
Colonial Metals Co  
De Bruyn Spectroscopic Solutions Ltd  
Coleshill Laboratories Ltd  
London & Scandinavian Met Co

Middlesbrough, England  
Sheffield, England  
Milperra, NSW, Australia  
Montreal, Canada  
Shanghai, China  
Luo Yang, He Nan, China  
Chennai, India  
Bangalore, India  
Mumbai, India  
Gliwice, Poland  
Columbia, PA, USA  
Johannesburg, South Africa  
Birmingham, England  
Rotherham, England

UKAS accreditation 0239  
UKAS accreditation 0012  
NATA accreditation 0492  
PRI accreditation 123077  
CNAL accreditation 0783  
CNAL accreditation 0173  
NABL accreditation 0025  
NABL accreditation 0371  
NABL accreditation 0367  
PCA accreditation AB274

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	ICP-MS	FAAS	OTHER
Lead	2-5, 7, 10, 12-14	8	1, 6, 9, 11	
Zinc	2, 4-6, 10-12	9	1, 3, 7, 8	
Iron	2-4, 9-12	5	1, 6-8	
Nickel	2-4, 7, 9-11, 14, 15	13	1, 6, 8, 12	5 photometric (dimethyl glyoxime)
Arsenic	1-6, 9, 13	10	7, 11, 12	8 photometric (turbidity)
Bismuth	1-8, 13	11	9, 10, 12	
Antimony	1, 3, 5-7, 10, 11, 13, 14	4	2, 8, 9	12 photometric (crystal violet)
Cobalt	1-3, 5, 7, 9, 10, 12-15	8	4, 6, 11	
Silver	1-3, 6, 9, 10, 12, 13	11	4, 5, 7, 8	
Phosphorus	3, 4, 6, 8, 10-13	-	-	2, 5, 7 photometric (molybdenum yellow) 1, 9 volumetric (alkalimetric)
Manganese	1, 3, 5, 6, 9-11	8	2, 4, 7	
Cadmium	1, 2, 4, 5, 8-12	7	3, 6	
Gold	1, 3, 4, 6-8, 10	2	5, 9	
Aluminium	1-4, 10, 11, 13-15	9	5, 7, 8	6, 12 photometric (chrome azurol S)
Tin	1, 3, 6-9, 11, 12	4	2, 5	10 photometric (phenyl fluorone)
Magnesium	1, 2, 4-9, 11, 15	13	3, 10, 12, 14	
Sulfur	4, 6, 10	-	-	others combustion (IR detection)
Selenium	1-3, 7-9, 11	6	4, 5, 10	
Tellurium	1, 3, 4, 6-8, 10-12	9	2, 5	
Indium	1-3, 7-10, 12, 12	6	4, 5, 11	

## 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, 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. This certification will therefore expire in December 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.