

# CERTIFICATE OF ANALYSIS

## 32X LB16 (batch A)

### Certified Reference Material Information

Type:	LEADED BRONZE (WROUGHT)
Form and Size:	Disc 32mm Diameter x 17mm Thickness
Manufactured by:	Copper Alloys Ltd
Certified and Supplied by:	MBH Analytical Ltd

### Assigned Values

#### Percentage element by weight

Element	Sn	Pb	Zn	Ni	Bi	Fe
Value <sup>1</sup>	5.55	18.78	0.450	0.793	0.0120	0.0040
Uncertainty <sup>2</sup>	0.04	0.18	0.008	0.011	0.0011	0.0005

Element	Al	Sb	Ag	P	S	Cu
Value <sup>1</sup>	(0.0012)	(0.0012)	0.0016	(0.0018)	0.0011	74.42
Uncertainty <sup>2</sup>	-	-	0.0003	-	0.0004	0.12

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 4th June 2009

C Eveleigh



## **Method of Preparation**

This reference material was produced from commercial-purity metals. The discs are the product of one batch which was induction melted, continuous-cast into bar of 42mm diameter and cold-drawn to the final dimension.

## **Sampling**

Samples for chemical analysis were taken from various positions throughout the batch. 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	Zn	Ni	Bi	Fe
1	5.469	18.63	0.429	0.769	0.0090	0.0025
2	5.50	18.64	0.437	0.770	0.0094	0.0027
3	5.505	18.66	0.447	0.771	0.0109	0.0034
4	5.527	18.70	0.447	0.777	0.0115	0.0034
5	5.556	18.79	0.450	0.788	0.0116	0.0036
6	5.562	18.80	0.451	0.794	0.0119	0.0040
7	5.565	18.81	0.455	0.798	0.0121	0.0044
8	5.580	18.93	0.456	0.803	0.0121	0.0045
9	5.60	19.10	0.463	0.805	0.0125	0.0046
10	5.601		0.463	0.813	0.013	0.0046
11	5.618			0.814	0.0140	0.0048
12				0.815	0.0155	0.0049
<b>Mean</b>	<b>5.55</b>	<b>18.78</b>	<b>0.450</b>	<b>0.793</b>	<b>0.0120</b>	<b>0.0040</b>
<b>Std Dev</b>	0.05	0.15	0.011	0.018	0.0018	0.0008
<b>C<sub>(95%)</sub></b>	0.03	0.12	0.008	0.011	0.0011	0.0005

Sample	Al	Sb	Ag	P	S	Cu
1	0.0001	0.0004	0.0010	0.0003	0.0005	74.28
2	0.0003	0.0006	0.0011	0.0009	0.0007	74.29
3	0.0008	0.0007	0.0011	0.0015	0.0007	74.30
4	0.0009	0.0011	0.0014	0.0017	0.0010	74.34
5	0.0013	0.0011	0.0017	0.0017	0.0011	74.49
6	0.0014	0.0011	0.0017	0.0018	0.0011	74.52
7	0.0015	0.0014	0.0018	0.0019	0.0011	74.55
8	0.0019	0.0014	0.0018	0.0022	0.0018	74.56
9	0.0020	0.0014	0.0021	0.0030	0.0020	
10	0.0020	0.0017	0.0022	0.0032		
11		0.0018				
12		0.0020				
<b>Mean</b>	<b>(0.0012)</b>	<b>(0.0012)</b>	<b>0.0016</b>	<b>(0.0018)</b>	<b>0.0011</b>	<b>74.42</b>
<b>Std Dev</b>	-	-	0.0004	-	0.0005	0.12
<b>C<sub>(95%)</sub></b>	-	-	0.0003	-	0.0004	0.10

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

Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation T0367
Shriram Institute for Industrial Research	Delhi, India	NABL accreditation T0045
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation T0371
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation T0025
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Colonial Metals Co	Columbia, PA, USA	
Coleshill Laboratories Ltd	Birmingham, England	

Note: to achieve the above accreditation (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		OTHER
Tin	1, 2, 5, 8, 10, 11	3, 7	4, 9	volumetric (iodate)
Lead	1, 4, 6, 8	7	6	photometric (phenylfluorone)
Zinc	1-5, 10	6-9	3, 9	volumetric (EDTA)
Nickel	1-3, 8-12	4-7	2, 5	electrogravimetric
Bismuth	1-3, 5, 6, 9-11	4, 7, 8, 12		
Iron	1, 2, 4, 5, 7, 9-11	3, 6, 8, 12		
Aluminium	1, 4, 5, 7, 8	2, 3, 10	6	photometric (chrome azurol S)
Antimony	2, 4-6, 8-11	1, 3, 7	9	ICP-MS
Silver	1, 2, 5-10	3, 4	12	ICP-MS
Phosphorus	1, 4-7, 10	-	2, 3, 9	photometric (molybdenum yellow)
Sulfur	2, 5, 8	-	8	ICP-MS
Copper	5, 6	-	1, 3, 4, 6, 7, 9	combustion with infra-red detection
			2, 8	volumetric (thiosulfate)
			1, 3, 4, 7	electrogravimetric

## Notes

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

This certification is applicable to the whole of the disc. However in accordance with normal practice for OES, it is appropriate to avoid use of the central area, of diameter ~8mm.

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 analysis. This certification will therefore expire in June 2029, 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.