MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

39X 17871 (batch D)

Certified Reference Material Information

Type: RESIDUALS IN COPPER (CHILL-CAST)

Form and Size: Disc ~40mm diameter

Produced by: Maybrey Reliance Ltd

Certified and supplied by: MBH Analytical Ltd

Assigned Values

Proportion of each element by weight, ug/g (ppm)

Element	Pb	Zn	Fe	Ni	Bi	Sb	Ag	Cd	Si
Value [1]	**503**	**918**	**19**	**412**	**527**	**141**	**309**	**27**	**14**
Uncertainty [2]	13	14	5	6	13	4	7	1	2

Element	As	P	Sn	Se	S	Te	Au	In	Ge
Value [1]	**150**	**204**	**1058**	**310**	**52**	**137**	**5**	**32**	**(9)**
Uncertainty [2]	6	10	23	6	4	8	1	4	-

Note: values in parentheses are not certified – they are provided for information only

Definitions

[1] 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.

[2] 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 28th December 2018

MBH ANALYTICAL LIMITED _____

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 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 batch. Approximately 10% 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. Multiple measurements were taken from each surface under test.

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, 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 according to the information 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	Pb	Zn	Fe	Ni	Bi	Sb	Ag	Cd	Si
1	0.0469	0.0891	0.0007	0.0393	0.0505	0.0127	0.0291	0.00247	0.0010
2	0.0482	0.0892	0.0008	0.0394	0.0507	0.0128	0.0293	0.0025	0.0011
3	0.0484	0.0901	0.00085	0.0396	0.0510	0.0132	0.0297	0.00258	0.00133
4	0.0486	0.0904	0.00116	0.0408	0.0516	0.0132	0.0298	0.0026	0.00140
5	0.0486	0.0921	0.00123	0.0411	0.0517	0.0138	0.0301	0.0026	0.0014
6	0.0488	0.0923	0.0015	0.0411	0.0522	0.0140	0.0308	0.0026	0.00143
7	0.0495	0.0926	0.00187	0.0413	0.0530	0.0141	0.0310	0.00263	0.0015
8	0.0499	0.0937	0.0020	0.0416	0.0533	0.0142	0.0312	0.0027	0.00177
9	0.0503	0.0940	0.0023	0.0417	0.0536	0.0143	0.0317	0.00277	
10	0.0509	0.0945	0.0024	0.0419	0.0557	0.0144	0.0317	0.00278	
11	0.0511		0.00266	0.0422	0.0564	0.0145	0.0319	0.0028	
12	0.0518		0.0027	0.0422		0.0146	0.0324	0.0028	
13	0.0531		0.0030	0.0422		0.0148	0.0327	0.0029	
14	0.0543		0.0031	0.0425		0.0149		0.0029	
15	0.0544					0.0151			
16						0.0154			
Mean	**0.0503**	**0.0918**	**0.00188**	**0.0412**	**0.0527**	**0.0141**	**0.0309**	**0.00269**	**0.00137**
Std Dev	0.0021	0.0020	0.00084	0.0011	0.0020	0.0008	0.0012	0.00014	0.00024
$C_{(95\%)}$	0.0013	0.0014	0.00049	0.0006	0.0013	0.0004	0.0007	0.00008	0.00020

Sample	As	P	Sn	Se	S	Te	Au	In	Ge
1	0.0129	0.0171	0.0985	0.0298	0.0038	0.0111	0.00030	0.0026	0.00088
2	0.0138	0.0180	0.0998	0.0301	0.0045	0.0124	0.00038	0.0026	0.00090
3	0.0142	0.0180	0.1010	0.0303	0.0045	0.0127	0.00043	0.0026	0.00090
4	0.0144	0.0188	0.1037	0.0304	0.0045	0.0129	0.00045	0.0028	0.00096
5	0.0145	0.0199	0.1040	0.0305	0.0046	0.0133	0.00050	0.0028	0.00098
6	0.0146	0.0201	0.1056	0.0310	0.0048	0.0135	0.00050	0.0030	<0.001
7	0.0147	0.0204	0.1065	0.0311	0.0050	0.0139	0.00050	0.0033	<0.001
8	0.0148	0.0205	0.1070	0.0314	0.0051	0.0141	0.00055	0.0038	<0.001
9	0.0148	0.0211	0.1076	0.0316	0.0053	0.0141	0.00060	0.0038	
10	0.0149	0.0211	0.1080	0.0322	0.0053	0.0143	0.00060	0.0039	
11	0.0150	0.0212	0.1081	0.0327	0.0057	0.0144	0.00063	0.0042	
12	0.0160	0.0213	0.1090		0.0057	0.0154			
13	0.0162	0.0216	0.1107		0.0061	0.0159			
14	0.0167	0.0229	0.1120		0.0063				
15	0.0170	0.0234			0.0066				
Mean	**0.0150**	**0.0204**	**0.1058**	**0.0310**	**0.0052**	**0.0137**	**0.00049**	**0.0032**	**0.00092**
Std Dev	0.0011	0.0018	0.0040	0.0009	0.0008	0.0013	0.00010	0.0006	0.00004
$C_{(95\%)}$	0.0006	0.0010	0.0023	0.0006	0.0004	0.0008	0.00007	0.0004	0.00005

The 95% confidence interval, $C_{(95\%)}$, is derived as follows:.

$$C_{(95\%)} = (t \times SD)/\sqrt{n}$$

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

Element Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Analytical Services Ltd	Sheffield, England	UKAS accreditation 0012
Anchorcert Analytical	Birmingham, England	UKAS accreditation 0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Shanghai Jinyi Test Technology Co	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Genitest, Inc	Montreal, Canada	PJ accreditation 95510
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Pvt Ltd	Mumbai, India	NABL Accreditation 0367
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
TEC-Eurolab SRL	Modena, Italy	Accredia accreditation 52
INCDMNR-IMNR	Pantelimon, Romania	
Mineral & Metallurgical Laboratories	Bangalore, India	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	
RR Intertek LSI	Rotterdam, Netherlands	

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
Lead	1-4, 7-12, 14	5, 6, 13	15	gravimetric (sulfate)
Zinc	2-7, 9, 10	1, 8		
Iron	1-5, 7-10, 12, 14	6, 11, 13		
Nickel	3-10, 13, 14	1, 2, 12	11	photometric (dimethyl glyoxime)
Bismuth	1, 2, 4, 5, 7-9, 11	3, 10	6	gravimetric
Antimony	1-10, 12, 15, 16	11, 14	13	volumetric (permanganate)
Silver	1, 2, 4, 5, 7, 9-11, 13	6, 8, 12	3	gravimetric (chloride)
Cadmium	1-3, 5-8, 11-14	4, 9, 10		
Silicon	1-3, 5-8	-	4	photometric (molybdenum blue)
Arsenic	1-9, 11-13, 15	10, 14		
Phosphorus	1, 3-5, 8, 10-14	-	2, 7, 15	photometric (molybdenum yellow)
			6, 9	volumetric (alkalimetric)
Tin	1-4, 6-9, 11, 13, 14	10, 12	5	volumetric (iodide)
Selenium	1, 3-9, 11	2, 10		
Sulfur	2, 5, 6, 8, 11	-	others	combustion (infra-red detection)
Tellurium	1-5, 7-13	6		
Gold	1-8, 10, 11	9		
Indium	2-7, 9, 10	1, 8, 11		
Germanium	1-8	-		

Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO 17034 and the associated Guides, 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 ~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 December 2038, 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.