

CERTIFICATE OF ANALYSIS

31X B21 (batch E)

Certified Reference Material Information

Type: LEADED 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	As	Si
Value ¹	0.101	0.113	29.55	0.126	0.0603	0.117	0.0908	0.059
Uncertainty ²	0.002	0.002	0.10	0.002	0.0009	0.002	0.0014	0.003

Element	P	Al	Bi	Sb	Te	S	Cu
Value ¹	0.1269	0.0244	0.104	0.105	0.0353	(0.002)	69.32
Uncertainty ²	0.0015	0.0008	0.005	0.002	0.0012	-	0.08

Note: values given 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:

MBH ANALYTICAL LIMITED _____

on 8th March 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

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 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, 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.

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	Fe	Mn	Ni	As	Si
1	0.0964	0.1082	29.32	0.1220	0.0580	0.1121	0.0871	0.0555
2	0.0970	0.1104	29.35	0.1230	0.0591	0.1130	0.0886	0.0560
3	0.0972	0.1110	29.40	0.1234	0.0591	0.1134	0.0887	0.0571
4	0.0974	0.1110	29.48	0.1237	0.0592	0.1146	0.0891	0.0573
5	0.0994	0.1112	29.51	0.1249	0.0595	0.1150	0.0891	0.0586
6	0.0998	0.1114	29.54	0.1250	0.0600	0.1160	0.0905	0.0589
7	0.0998	0.1131	29.56	0.1255	0.0601	0.1165	0.0907	0.0598
8	0.0999	0.1143	29.58	0.1263	0.0602	0.1169	0.0911	0.0598
9	0.1001	0.1150	29.59	0.1264	0.0603	0.1175	0.0912	0.0609
10	0.1011	0.1150	29.60	0.1270	0.0606	0.1180	0.0913	0.0631
11	0.1015	0.1150	29.61	0.1277	0.0608	0.1190	0.0916	0.0633
12	0.1017	0.1151	29.66	0.1290	0.0612	0.1194	0.0920	
13	0.1026	0.1157	29.69	0.1290	0.0615	0.1197	0.0922	
14	0.1040	0.1160	29.75	0.1301	0.0618	0.1200	0.0930	
15	0.1050	0.1170		0.1305	0.0620	0.1200	0.0951	
16	0.1072				0.0621	0.1205		
Mean	0.1006	0.1133	29.55	0.1262	0.0603	0.1170	0.0908	0.0591
Std Dev	0.0030	0.0026	0.12	0.0026	0.0012	0.0027	0.0020	0.0026
C_(95%)	0.0016	0.0014	0.07	0.0015	0.0006	0.0015	0.0011	0.0018

Sample	P	Al	Bi	Sb	Te	S	Cu
1	0.1240	0.0223	0.1003	0.1010	0.0327	0.0010	69.19
2	0.1253	0.0227	0.1010	0.1014	0.0336	0.0011	69.25
3	0.1260	0.0228	0.1014	0.1017	0.0344	0.0011	69.27
4	0.1260	0.0230	0.1014	0.1030	0.0347	0.0015	69.29
5	0.1260	0.0232	0.1019	0.1032	0.0351	0.0015	69.30
6	0.1261	0.0240	0.1030	0.1035	0.0352	0.0019	69.32
7	0.1264	0.0244	0.1041	0.1038	0.0355	0.0019	69.32
8	0.1265	0.0245	0.1044	0.1050	0.0355	0.0021	69.37
9	0.1270	0.0246	0.1070	0.1050	0.0356	0.0022	69.37
10	0.1285	0.0246	0.1070	0.1050	0.0363	0.0022	69.40
11	0.1288	0.0255	0.1080	0.1071	0.0364	0.0025	69.48
12	0.1288	0.0255		0.1080	0.0387		
13	0.1300	0.0257		0.1080			
14		0.0262		0.1081			
15		0.0266					
Mean	0.1269	0.0244	0.1036	0.1046	0.0353	(0.0017)	69.32
Std Dev	0.0017	0.0014	0.0027	0.0025	0.0015	-	0.08
C_(95%)	0.0010	0.0008	0.0018	0.0014	0.0010	-	0.05

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
Shanghai Jinyi Test Technology Co
Luo Yang Copper
Genitest, Inc
Bureau Veritas CPS Pvt Ltd
Raghavendra Spectromet Laboratory
Institute of Non-Ferrous Metals
Tec-Eurolab
Microlab
Mineral & Metallurgical Laboratories
Colonial Metals Co
AMG Superalloys UK Ltd
Coleshill Laboratories Ltd
Analyticka Laborator Lithea sro

Middlesbrough, England
Sheffield, England
Milperra, NSW, Australia
Shanghai, China
Luo Yang, He Nan, China
Montreal, Canada
Chennai, India
Bangalore, India
Gliwice, Poland
Campogalliano, Italy
Chennai, India
Bangalore, India
Columbia, PA, USA
Rotherham, England
Birmingham, England
Brno, Czech Republic

UKAS accreditation 0239
UKAS accreditation 0012
NATA accreditation 0492
CNAL accreditation 0783
CNAL accreditation 0173
PRI accreditation 123077
NABL accreditation 0025
NABL accreditation 0371
PCA accreditation AB274
ACCREDIA accreditation 52

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	XRF	FAAS		OTHER
Tin	1-3, 6, 8, 9, 11-14, 16	-	4, 7, 10	5	photometric (phenyl fluorone)
Lead	1, 2, 6-10, 13, 14	-	3, 5, 11, 12	15	volumetric (iodate)
				4	gravimetric (sulfate)
Zinc	1, 2, 12, 13	4	-	15	electrogravimetric
				3, 5-11, 14	volumetric (EDTA)
Iron	1, 3, 4, 7, 9-11, 13-15	12	5, 8	2	photometric (orthophenanthroline)
				6	volumetric (dichromate)
Manganese	1-5, 7-9, 11, 13, 14, 16	-	6, 10, 12, 16	15	volumetric (arsenite)
Nickel	1-4, 6, 8, 10, 12-14, 16	11	7, 9, 15	5	photometric (dimethyl glyoxime)
Arsenic	1, 2, 4-6, 8-11, 13-15	-	3, 7	12	photometric (turbidity)
Silicon	4, 6-8, 11	-	-	1, 2, 5	photometric (molybdenum blue)
				3, 9, 10	gravimetric (perchloric acid)
Phosphorus	4, 7-12	-	-	1, 3, 13	photometric (molybdenum yellow)
				2, 5, 6	volumetric (alkalimetric)
Aluminium	1, 4, 6-10, 12-15	-	2, 5	3	photometric (chrome azurol-S)
				11	volumetric (EDTA)
Bismuth	1, 3-10	-	11	2	photometric (iodide)
Antimony	1, 2, 5-7, 10-12, 14	8	3, 13	4	volumetric (permanganate)
				9	photometric (crystal violet)
Tellurium	1, 3-12	-	2		
Sulfur	7, 8, 11	-	-	1-6, 9, 10	combustion (infra-red detection)
Copper	1, 8	-	-	2, 6, 7	volumetric (thiosulfate)
				3-5, 9-11	electrogravimetric

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 10mm. Material to the rear of the disc, to a depth of ~5mm, 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 March 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.