

CERTIFICATE OF ANALYSIS

39X 17868 (batch AH)

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	Mn	Bi	Sb	Cd
Value ¹	0.0190	0.55	0.0227	0.0277	0.0204	0.0226	0.0319	0.0280
Uncertainty ²	0.0006	0.02	0.0006	0.0014	0.0010	0.0010	0.0008	0.0008

Element	Co	Ag	Si	As	P	Sn	Se
Value ¹	0.0050	0.0209	0.0105	0.0145	0.0107	0.0322	0.0103
Uncertainty ²	0.0005	0.0004	0.0004	0.0009	0.0006	0.0009	0.0005

Element	S	Al	Cr	Te	In	Mg	Ge
Value ¹	0.0124	0.0098	0.0267	0.0273	0.0197	(0.010)	0.0272
Uncertainty ²	0.0008	0.0006	0.0008	0.0004	0.0005	-	0.0009

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 June 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. At least 15% 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 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.

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	Mn	Bi	Sb	Cd
1	0.0170	0.5405	0.0209	0.0255	0.0185	0.0212	0.0288	0.0258
2	0.0172	0.5436	0.0214	0.0268	0.0192	0.0213	0.0301	0.0261
3	0.0180	0.5465	0.0215	0.0268	0.0193	0.0214	0.0312	0.0261
4	0.0184	0.5482	0.0217	0.0270	0.0198	0.0214	0.0313	0.0263
5	0.0185	0.5490	0.0220	0.0273	0.0199	0.0222	0.0315	0.0267
6	0.0187	0.5498	0.0220	0.0273	0.0201	0.0224	0.0317	0.0277
7	0.0188	0.5530	0.0223	0.0273	0.0202	0.0227	0.0317	0.0278
8	0.0189	0.5530	0.0226	0.0275	0.0204	0.0230	0.0322	0.0278
9	0.0189	0.5537	0.0228	0.0277	0.0204	0.0230	0.0322	0.0281
10	0.0193	0.5550	0.0230	0.0281	0.0204	0.0232	0.0324	0.0287
11	0.0193	0.5580	0.0231	0.0282	0.0204	0.0233	0.0326	0.0288
12	0.0195	0.5610	0.0231	0.0291	0.0207	0.0234	0.0327	0.0289
13	0.0197	0.5650	0.0232	0.0292	0.0209	0.0237	0.0333	0.0290
14	0.0197	0.5683	0.0232	0.0296	0.0211	0.0240	0.0334	0.0294
15	0.0200		0.0239		0.0215		0.0345	0.0296
16	0.0204		0.0240		0.0215			0.0298
17	0.0211		0.0246		0.0223			0.0299
Mean	0.0190	0.5532	0.0227	0.0277	0.0204	0.0226	0.0319	0.0280
Std Dev	0.0011	0.0079	0.0010	0.0011	0.0009	0.0009	0.0013	0.0014
C_(95%)	0.0005	0.0045	0.0005	0.0006	0.0005	0.0005	0.0007	0.0007

Sample	Co	Ag	Si	As	P	Sn	Se
1	0.0042	0.0198	0.0091	0.0117	0.0094	0.0303	0.0083
2	0.0042	0.0200	0.0098	0.0129	0.0099	0.0305	0.0088
3	0.0042	0.0200	0.0099	0.0132	0.0100	0.0313	0.0097
4	0.0045	0.0203	0.0100	0.0133	0.0100	0.0314	0.0099
5	0.0047	0.0205	0.0103	0.0135	0.0102	0.0317	0.0100
6	0.0048	0.0208	0.0103	0.0140	0.0104	0.0321	0.0101
7	0.0048	0.0211	0.0106	0.0141	0.0105	0.0322	0.0104
8	0.0051	0.0212	0.0108	0.0144	0.0105	0.0324	0.0104
9	0.0051	0.0213	0.0108	0.0144	0.0107	0.0328	0.0105
10	0.0053	0.0214	0.0109	0.0155	0.0107	0.0328	0.0106
11	0.0053	0.0215	0.0110	0.0157	0.0107	0.0329	0.0107
12	0.0053	0.0216	0.0112	0.0162	0.0109	0.0331	0.0108
13	0.0054	0.0220	0.0115	0.0165	0.0119	0.0339	0.0113
14	0.0055			0.0171	0.0121	0.0340	0.0115
15	0.0062				0.0121		0.0119
Mean	0.0050	0.0209	0.0105	0.0145	0.0107	0.0322	0.0103
Std Dev	0.0006	0.0007	0.0007	0.0015	0.0008	0.0011	0.0009
C_(95%)	0.0003	0.0004	0.0004	0.0009	0.0004	0.0006	0.0005

Sample	S	Al	Cr	Te	In	Mg	Ge
1	0.0107	0.0076	0.0239	0.0260	0.0175	0.0077	0.0255
2	0.0111	0.0086	0.0249	0.0265	0.0189	0.0085	0.0255
3	0.0115	0.0088	0.0254	0.0269	0.0191	0.0087	0.0256
4	0.0120	0.0091	0.0258	0.0271	0.0192	0.0092	0.0263
5	0.0122	0.0091	0.0261	0.0271	0.0193	0.0092	0.0269
6	0.0123	0.0094	0.0262	0.0273	0.0194	0.0093	0.0270
7	0.0124	0.0097	0.0263	0.0275	0.0195	0.0094	0.0276
8	0.0125	0.0101	0.0268	0.0276	0.0195	0.0098	0.0279
9	0.0126	0.0109	0.0271	0.0276	0.0196	0.0103	0.0287
10	0.0126	0.0117	0.0275	0.0278	0.0198	0.0106	0.0288
11	0.0129	0.0128	0.0277	0.0280	0.0200	0.0112	0.0289
12	0.0132		0.0278	0.0280	0.0204	0.0114	
13	0.0133		0.0282		0.0206		
14	0.0145		0.0283		0.0211		
15			0.0283		0.0217		
Mean	0.0124	0.0098	0.0267	0.0273	0.0197	0.0096	0.0272
Std Dev	0.0010	0.0015	0.0013	0.0006	0.0011	0.0011	0.0013
C_(95%)	0.0005	0.0006	0.0007	0.0004	0.0005	0.0007	0.0009

Note: For the definition and derivation of C_(95%), see page 2.

Participating Laboratories

Exova Ltd	Middlesbrough, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
AnchorCert Analytical	Birmingham, UK	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
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation	1461
TCR Engineering Services PVT. Ltd	Mumbai, India	NABL accreditation	0367
Raghavendra Spectro Metallurgical Laboratory	Bangalore, India	NABL accreditation	0371
Institute of Non-ferrous Metals	Gliwice, Poland	PCA accreditation	AB274
TEC Eurolab SRL	Modena, Italy	ACCREDIA accreditation	52
AMG Superalloys UK Ltd	Rotherham, UK		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
INCDMNR-IMNR	Pantelimon, Romania		
RR Intertek LSI	Rotterdam, Netherlands		
Mineral & Metallurgical Laboratories	Bangalore, India		

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
Lead	1, 3-5, 7, 8, 10-13, 16, 17	2, 9, 14, 15	6 gravimetric (sulfate)
Zinc	1-8, 12	9, 11, 13, 14	10 volumetric (EDTA)
Iron	1, 3-10, 14, 16, 17	2, 12, 15	11, 13 photometric (orthophenanthroline)
Nickel	1-4, 6-8, 12, 13	5, 10, 11	9, 14 photometric (dimethyl glyoxime)
Manganese	3-7, 10-14, 16, 17	1, 2, 9, 15	8 volumetric (arsenite)
Bismuth	1, 3-6, 10, 12-14	2, 7, 8	9, 11 photometric (iodide)
Antimony	1-5, 8, 9, 11-14	6, 15	7, 10 volumetric (permanganate)
Cadmium	1-3, 5-10, 13, 14, 16	4, 12, 15, 17	11 gravimetric
Cobalt	3, 5-7, 9-15	1, 2, 4, 8	
Silver	1, 3-5, 8-10, 12	2, 7, 11, 13	6 gravimetric (chloride)
Silicon	1, 3, 4, 6-8, 10, 12, 13	-	2, 5, 9, 11 photometric (molybdenum yellow)
Arsenic	2-4, 6-14	1, 5	
Phosphorus	2-8, 10, 11, 14	-	1, 12, 13 photometric (molybdenum yellow)
			9, 15 volumetric (alkalimetric)
Tin	1, 3-5, 8-14	2	6, 7 photometric (phenyl fluorone)
Selenium	1-13, 15	14	
Sulfur	4, 7, 9, 12	-	others combustion (IR or volumetric detection)
Aluminium	3-7, 9, 11	2, 10	1, 8 photometric (chrome azurol S)
Chromium	1, 2, 4, 5, 8-11, 13-15	3, 6, 7	12 volumetric (arsenite)
Tellurium	1-7, 9-12	8	
Indium	1-3, 5-15	4	
Magnesium	1-3, 5-9	4, 10-12	
Germanium	1-11	-	

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, 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. 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 June 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.