

CERTIFICATE OF ANALYSIS

39X 27866 (A)

Certified Reference Material Information

Type: RESIDUALS IN COPPER (WROUGHT)
Form and Size: Disc ~38mm Diameter x 20mm Thickness
Produced by: Copper Alloys Ltd
Certified and supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Pb	Zn	Sn	Fe	Ni	As	Sb
Value ¹	0.0054	0.0287	0.0448	0.0030	0.0487	0.0383	0.0052
Uncertainty ²	0.0005	0.0009	0.0012	0.0004	0.0005	0.0006	0.0003

Element	Co	Cd	S	Se	Ag	Ge
Value ¹	0.0308	0.0139	0.0469	0.0028	0.0057	0.0029
Uncertainty ²	0.0007	0.0003	0.0014	0.0003	0.0005	0.0004

Element	Bi	Cr	P	Au	In	Te
Value ¹	0.0047	0.0012	0.0147	0.0016	0.0437	0.0032
Uncertainty ²	0.0003	0.0002	0.0010	0.0002	0.0012	0.0004

Note: ^{1,2}: for definitions, see page 2

Certified by:

MBH ANALYTICAL LIMITED _____

on 20th August 2010

C Eveleigh



Method of Preparation

This reference material was produced from commercial-purity copper, and a combination of pure elements and binaries. Discs were produced by casting into a single ingot, which was hot extruded to the final diameter.

Sampling

Milled samples for chemical analysis, and discs for homogeneity checks, were taken randomly from several positions throughout the batch. At least 5% of all discs were selected for homogeneity checking.

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.

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.

Definitions

- ¹ The assigned 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, listed on page 3.
- ² The analytical uncertainty values are generated from the 95% half-width confidence interval $C_{(95\%)}$, which is derived from the wet analysis results, in accordance with the following 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.

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 above. 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, also 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.

Usage

Intended use: With optical emission and X-ray fluorescence spectrometers.

Recommended method of use: Copper and its alloys are generally prepared by machining on a mill or 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 optimise precision and accuracy. 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	Sn	Fe	Ni	As	Sb
1	0.0044	0.0263	0.0420	0.0019	0.0474	0.0371	0.0046
2	0.0046	0.0269	0.0428	0.0021	0.0477	0.0372	0.0046
3	0.0048	0.0270	0.0430	0.0022	0.0480	0.0372	0.0046
4	0.0049	0.0278	0.0440	0.0027	0.0480	0.0375	0.0046
5	0.0050	0.0280	0.0443	0.0028	0.0481	0.0379	0.0047
6	0.0051	0.0285	0.0443	0.0030	0.0481	0.0380	0.0048
7	0.0052	0.0285	0.0445	0.0031	0.0485	0.0380	0.0053
8	0.0053	0.0286	0.0452	0.0031	0.0487	0.0381	0.0053
9	0.0059	0.0287	0.0453	0.0032	0.0490	0.0386	0.0053
10	0.0063	0.0289	0.0456	0.0034	0.0492	0.0389	0.0054
11	0.0065	0.0290	0.0460	0.0035	0.0495	0.0389	0.0055
12	0.0066	0.0304	0.0475	0.0036	0.0498	0.0391	0.0057
13		0.0313	0.0483	0.0038	0.0498	0.0398	0.0058
14		0.0319		0.0041	0.0501	0.0400	0.0061
Mean	0.0054	0.0287	0.0448	0.0030	0.0487	0.0383	0.0052
Std Dev	0.0008	0.0016	0.0018	0.0006	0.0009	0.0009	0.0005
C_(95%)	0.0005	0.0009	0.0011	0.0004	0.0005	0.0005	0.0003

Sample	Co	Cd	S	Se	Ag	Ge
1	0.0291	0.0128	0.0445	0.0021	0.0050	0.0023
2	0.0293	0.0131	0.0447	0.0022	0.0051	0.0026
3	0.0299	0.0135	0.0448	0.0023	0.0052	0.0028
4	0.0304	0.0139	0.0454	0.0025	0.0052	0.0029
5	0.0310	0.0139	0.0455	0.0025	0.0053	0.0031
6	0.0310	0.0139	0.0455	0.0027	0.0053	0.0034
7	0.0312	0.0140	0.0458	0.0028	0.0057	0.0035
8	0.0313	0.0141	0.0490	0.0029	0.0058	
9	0.0315	0.0142	0.0490	0.0029	0.0059	
10	0.0316	0.0142	0.0491	0.0031	0.0061	
11		0.0143	0.0492	0.0032	0.0063	
12		0.0144	0.0498	0.0033	0.0067	
13		0.0147		0.0034	0.0069	
Mean	0.0308	0.0139	0.0469	0.0028	0.0057	0.0029
Std Dev	0.0010	0.0005	0.0021	0.0004	0.0006	0.0004
C_(95%)	0.0007	0.0003	0.0014	0.0003	0.0004	0.0004

Sample	Bi	Cr	P	Au	In	Te
1	0.0038	0.0008	0.0118	0.0012	0.0411	0.0026
2	0.0039	0.0009	0.0128	0.0012	0.0411	0.0027
3	0.0041	0.0010	0.0136	0.0013	0.0412	0.0027
4	0.0042	0.0011	0.0144	0.0014	0.0416	0.0028
5	0.0044	0.0012	0.0145	0.0015	0.0419	0.0030
6	0.0047	0.0012	0.0147	0.0015	0.0436	0.0030
7	0.0047	0.0012	0.0148	0.0015	0.0438	0.0030
8	0.0048	0.0013	0.0150	0.0016	0.0441	0.0036
9	0.0049	0.0014	0.0150	0.0017	0.0445	0.0039
10	0.0050	0.0015	0.0151	0.0017	0.0447	0.0039
11	0.0050	0.0015	0.0169	0.0018	0.0451	0.0042
12	0.0051		0.0172	0.0018	0.0455	
13	0.0052			0.0019	0.0461	
14	0.0054			0.0019	0.0478	
15	0.0055					
Mean	0.0047	0.0012	0.0147	0.0016	0.0437	0.0032
Std Dev	0.0005	0.0002	0.0015	0.0002	0.0021	0.0006
C_(95%)	0.0003	0.0002	0.0010	0.0001	0.0012	0.0004

For the definition of C_(95%) see page 2.

Participating Laboratories

Exova Materials Testing Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing Inc	Hatfield, PA, USA	A2LA accreditation 0117
Anderson Laboratories	Greendale, WI, USA	A2LA accreditation 0711
Genitest Inc	Montreal, Canada	PRI accreditation 123077
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation T025
TCR Engineering Servs Pvt Ltd	Mumbai, India	NABL accreditation T367
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
De Bruyn Spectroscopic Solutions	Johannesburg, South Africa	
London & Scandinavian Met. Co	Rotherham, England	
Colehill Laboratories Ltd	Birmingham, England	
Evans Analytical Group, Shiva Technologies	Syracuse, NY, USA	

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	GD-MS	FAAS	OTHER	
Lead	1, 2, 4, 5, 9, 10, 12	7	3, 11	6, 8	volumetric (EDTA)
Zinc	1-3, 5, 9-12, 14	-	6, 8, 13	4, 7	volumetric (EDTA)
Tin	1, 2, 4, 5, 7-9, 11, 12	3	6, 10	13	photometric (phenyl fluorone)
Iron	2, 5-8, 10-13	14	3, 4, 9	1	photometric (orthophenanthroline)
Nickel	1-3, 5-8, 11, 12	9	4, 10, 13, 14		
Arsenic	1-4, 6, 7, 9, 11-13	14	8, 10	5	photometric (turbidity)
Antimony	2-5, 8-13	1	6, 7, 14		
Cobalt	1, 2, 4, 6-9	-	3, 5, 10		
Cadmium	1, 2, 5-9, 11, 13	-	3, 4, 10, 12		
Sulfur	5, 10, 11	9	-	1, 6 others	combustion (volumetric detection) combustion (infra-red detection)
Selenium	1, 5, 7-9, 11-13	6	3, 10	2 4	AAS-hydride ICP-MS
Silver	1, 2, 4-6, 10-13	9	3, 7, 8		
Germanium	2, 4-7	3	-	1	ICP-MS
Bismuth	1-3, 5-9, 11, 12, 14	4	10, 13, 15		
Chromium	1-3, 6-9	-	4, 5, 10, 11		
Phosphorus	1, 2, 5-7, 11, 12	9	-	3, 4, 8 10	photometric (molybdenum blue) volumetric (alkalimetric)
Gold	1, 2, 4, 6-10, 12-14	11	5	3	ICP-MS
Indium	1-4, 6, 7, 9-13	-	5, 8, 14		
Tellurium	1-3, 6-9,	10	5, 11	4	ICP-MS

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 above certification is applicable to all of the disc. However, in accordance with normal practice for OES sampling, it is appropriate to avoid use of the central portion of ~6mm diameter.

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 40 years from the date of original analysis. This certification will therefore expire in August 2050, 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.