

# CERTIFICATE OF ANALYSIS

**36X CN9 (batch K)**

## Certified Reference Material Information

Type: CUPRO-NICKEL (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	Sn	Pb	Fe	Ni	Co	Si	Mn	Cr	Al
Value <sup>1</sup>	0.0351	0.070	1.262	26.40	0.0785	0.406	1.005	2.02	0.051
Uncertainty <sup>2</sup>	0.0008	0.003	0.010	0.06	0.0012	0.006	0.014	0.02	0.002

Element	Nb	P	S	C	Bi	Zr	Ti	Cu
Value <sup>1</sup>	1.37	(0.021)	0.0105	0.005	0.0197	0.102	0.111	66.94
Uncertainty <sup>2</sup>	0.02	-	0.0005	0.001	0.0007	0.003	0.002	0.06

Note: values given in parentheses are not certified - they are provided for information only.

## Definitions

- <sup>1</sup> 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, 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 4<sup>th</sup> January 2017

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## **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 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. The testing procedure was in accordance with ASTM E826 and the material found acceptable.

From this test data, through-batch variation values were derived for each element as an indicator of any minor compositional variation (as determined for the specific sample size and other limitations of 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.

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	Sn	Pb	Fe	Ni	Co	Si	Mn	Cr	Al
1	0.0326	0.0660	1.251	26.330	0.0748	0.3896	0.974	1.989	0.0487
2	0.0329	0.0660	1.251	26.365	0.0757	0.3920	0.975	1.991	0.0498
3	0.0340	0.0662	1.252	26.369	0.0765	0.3950	0.978	2.000	0.0500
4	0.0340	0.0665	1.255	26.390	0.0769	0.3980	0.998	2.003	0.0501
5	0.0348	0.0681	1.257	26.390	0.0772	0.3996	1.001	2.007	0.0502
6	0.0348	0.0685	1.263	26.392	0.0775	0.4070	1.002	2.016	0.0504
7	0.0351	0.0699	1.263	26.420	0.0776	0.4084	1.003	2.025	0.0504
8	0.0355	0.0701	1.273	26.422	0.0785	0.4084	1.004	2.025	0.0505
9	0.0355	0.0708	1.276	26.440	0.0787	0.4110	1.009	2.033	0.0525
10	0.0358	0.0717	1.278	26.450	0.0795	0.4124	1.010	2.033	0.0525
11	0.0359	0.0740		26.463	0.0796	0.4132	1.022	2.059	0.0532
12	0.0362	0.0744			0.0799	0.4146	1.025	2.063	0.0548
13	0.0362	0.0745			0.0803	0.4155	1.037		
14	0.0363	0.0760			0.0807	0.4200	1.038		
15	0.0363				0.0811				
16					0.0813				
<b>Mean</b>	<b>0.0351</b>	<b>0.0702</b>	<b>1.262</b>	<b>26.403</b>	<b>0.0785</b>	<b>0.4061</b>	<b>1.005</b>	<b>2.020</b>	<b>0.0511</b>
<b>Std Dev</b>	0.0012	0.0035	0.010	0.040	0.0020	0.0095	0.021	0.024	0.0018
<b>C<sub>(95%)</sub></b>	0.0007	0.0020	0.007	0.027	0.0011	0.0055	0.012	0.015	0.0011

Sample	Nb	P	S	C	Bi	Zr	Ti	Cu
1	1.323	0.0200	0.0090	0.0041	0.0180	0.0996	0.1060	66.860
2	1.340	0.0201	0.0098	0.0041	0.0188	0.0999	0.1079	66.880
3	1.345	0.0203	0.0100	0.0046	0.0192	0.1010	0.1090	66.902
4	1.350	0.0204	0.0101	0.0047	0.0196	0.1032	0.1090	66.906
5	1.353	0.0211	0.0102	0.0050	0.0196	0.1042	0.1092	66.940
6	1.353	0.0219	0.0103	0.0053	0.0196	0.1047	0.1101	66.943
7	1.373	0.0221	0.0103		0.0198		0.1103	66.947
8	1.395		0.0108		0.0198		0.1108	66.950
9	1.401		0.0109		0.0199		0.1110	66.990
10	1.411		0.0110		0.0200		0.1130	66.990
11	1.415		0.0114		0.0212		0.1137	67.000
12			0.0116		0.0214		0.1140	
13							0.1140	
<b>Mean</b>	<b>1.369</b>	<b>0.0208</b>	<b>0.0104</b>	<b>0.0046</b>	<b>0.0197</b>	<b>0.1021</b>	<b>0.1106</b>	<b>66.937</b>
<b>Std Dev</b>	0.032	0.0009	0.0007	0.0005	0.0009	0.0022	0.0025	0.046
<b>C<sub>(95%)</sub></b>	0.021	0.0008	0.0005	0.0005	0.0006	0.0023	0.0015	0.031

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

Exova Ltd	Middlesbrough, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
Metals Technology Testing Ltd	Sheffield, UK	UKAS accreditation	0963
AnchorCert Analytical	Birmingham, UK	UKAS accreditation	0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation	0492
Genitest Inc.	Montreal, QC, Canada	PRI accreditation	123077
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation	0173
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation	1461
Bureau Veritas CPS Ltd	Chennai, India	NABL accreditation	0025
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
Mineral and Metallurgical Laboratories	Bangalore, India		
AMG Superalloys UK Ltd	Rotherham, UK		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
Coleshill Laboratories Ltd	Coleshill, UK		

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
Tin	2-7, 9-11, 14, 15	1, 13	8	volumetric (iodine)
			12	photometric (phenyl fluorone)
Lead	1, 2, 5, 8, 10-14	3, 4, 6, 9	7	gravimetric
Iron	1, 4-6, 8, 10	2	3	volumetric (permanganate)
			7, 9	photometric (orthophenanthroline)
Nickel	2, 3, 7, 9	-	1, 5, 6, 8, 10	gravimetric (dimethyl glyoxime)
			4	photometric (dimethyl glyoxime)
			11	volumetric (dimethyl glyoxime, EDTA)
Cobalt	3-7, 9-13, 16	1, 2, 8, 14	15	gravimetric (oxide)
Silicon	1-3, 7, 8, 10, 13	-	4, 14	photometric (molybdenum blue)
			5, 6, 9, 11-12	gravimetric (perchloric acid)
Manganese	4-8, 10, 13, 14	1, 3, 12	2, 9	photometric (periodate)
			11	volumetric (arsenite)
Chromium	2, 3, 5, 6, 8, 11, 12	1, 4, 7	9, 10	volumetric (ferrous ammonium sulfate)
Aluminium	1, 3-6, 8-12	7	2	volumetric (EDTA)
Niobium	1, 2, 5-9, 11	3, 4	10	gravimetric
Phosphorus	1, 6, 7	-	2, 5	photometric (molybdenum blue)
			3, 4	volumetric (alkalimetric)
Sulfur	2, 8	-	1, 3-7, 9-12	combustion (IR or volumetric detection)
Carbon	-	-	1-6	combustion (infra-red detection)
Bismuth	2, 4-9, 10, 11	1, 10	3	photometric (iodide)
Zirconium	2, 4-6	1, 3		
Titanium	1-8, 11, 13	10	9, 12	photometric (peroxide, DAP)
Copper	1, 4, 6-8, 10	-	2, 3	electrogravimetric
			5, 9, 11	volumetric (thiosulfate)

## 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. 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 January 2037, although we reserve the right to make changes as issue revisions, in the intervening period.

The manufacture, analysis and certification of this product were supervised by L Maxim, Technical Director, MBH Analytical Ltd.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.