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# CERTIFICATE OF ANALYSIS

## 32X SN4 (batch B)

## Certified Reference Material Information

| | |
|---|---|
| Type: | BRONZE (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 | Ni | Al | As | P |
|---|---|---|---|---|---|---|---|---|
| **Value** [1] | **18.96** | **0.864** | **0.496** | **0.0811** | **0.607** | **0.0513** | **0.0651** | **1.208** |
| Uncertainty [2] | 0.06 | 0.007 | 0.007 | 0.0011 | 0.007 | 0.0007 | 0.0010 | 0.017 |

| Element | Mn | Co | Bi | Sb | Si | Ag | S | Cu |
|---|---|---|---|---|---|---|---|---|
| **Value** [1] | **0.0148** | **0.100** | **0.0150** | **0.143** | **0.0223** | **0.495** | **0.0132** | **76.87** |
| Uncertainty [2] | 0.0002 | 0.002 | 0.0003 | 0.004 | 0.0008 | 0.008 | 0.0007 | 0.11 |

## 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 23rd July 2017

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 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

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, 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, 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.<br><br>Preparation should be the same for reference materials and the samples for test.<br><br>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 | Ni | Al | As | P |
|---|---|---|---|---|---|---|---|---|
| 1 | 18.81 | 0.8442 | 0.4749 | 0.0780 | 0.5925 | 0.0489 | 0.0625 | 1.172 |
| 2 | 18.87 | 0.8530 | 0.4790 | 0.0798 | 0.5930 | 0.0498 | 0.0626 | 1.181 |
| 3 | 18.90 | 0.8550 | 0.4830 | 0.0803 | 0.5943 | 0.0506 | 0.0639 | 1.189 |
| 4 | 18.90 | 0.8590 | 0.4920 | 0.0804 | 0.6075 | 0.0506 | 0.0640 | 1.201 |
| 5 | 18.93 | 0.8597 | 0.4940 | 0.0805 | 0.6080 | 0.0508 | 0.0647 | 1.201 |
| 6 | 18.98 | 0.8655 | 0.4953 | 0.0806 | 0.6080 | 0.0510 | 0.0653 | 1.201 |
| 7 | 19.00 | 0.8660 | 0.4990 | 0.0808 | 0.6120 | 0.0512 | 0.0654 | 1.215 |
| 8 | 19.00 | 0.8669 | 0.4995 | 0.0809 | 0.6145 | 0.0513 | 0.0658 | 1.217 |
| 9 | 19.03 | 0.8712 | 0.5000 | 0.0819 | 0.6150 | 0.0514 | 0.0667 | 1.235 |
| 10 | 19.03 | 0.8719 | 0.5051 | 0.0822 | 0.6152 | 0.0515 | 0.0668 | 1.239 |
| 11 | 19.04 | 0.8760 | 0.5080 | 0.0822 | 0.6200 | 0.0523 | 0.0669 | 1.240 |
| 12 | 19.07 | 0.8800 | 0.5108 | 0.0834 | | 0.0525 | 0.0671 | |
| 13 | | | 0.5110 | 0.0834 | | 0.0529 | | |
| 14 | | | | | | 0.0538 | | |
| **Mean** | **18.96** | **0.8640** | **0.4963** | **0.0811** | **0.6073** | **0.0513** | **0.0651** | **1.208** |
| **Std Dev** | 0.08 | 0.0103 | 0.0117 | 0.0015 | 0.0097 | 0.0013 | 0.0016 | 0.023 |
| **$C_{(95\%)}$** | 0.05 | 0.0065 | 0.0070 | 0.0009 | 0.0066 | 0.0007 | 0.0010 | 0.016 |

| Sample | Mn | Co | Bi | Sb | Si | Ag | S | Cu |
|---|---|---|---|---|---|---|---|---|
| 1 | 0.0143 | 0.0933 | 0.0141 | 0.1330 | 0.0199 | 0.4734 | 0.0111 | 76.63 |
| 2 | 0.0143 | 0.0938 | 0.0144 | 0.1340 | 0.0205 | 0.4740 | 0.0113 | 76.64 |
| 3 | 0.0144 | 0.0965 | 0.0144 | 0.1353 | 0.0206 | 0.4773 | 0.0122 | 76.71 |
| 4 | 0.0144 | 0.0975 | 0.0145 | 0.1360 | 0.0214 | 0.4857 | 0.0122 | 76.81 |
| 5 | 0.0146 | 0.0984 | 0.0146 | 0.1360 | 0.0217 | 0.4911 | 0.0129 | 76.83 |
| 6 | 0.0148 | 0.0988 | 0.0148 | 0.1403 | 0.0219 | 0.4948 | 0.0130 | 76.83 |
| 7 | 0.0148 | 0.1010 | 0.0149 | 0.1448 | 0.0222 | 0.4970 | 0.0133 | 76.85 |
| 8 | 0.0148 | 0.1018 | 0.0150 | 0.1455 | 0.0225 | 0.4995 | 0.0135 | 76.90 |
| 9 | 0.0150 | 0.1018 | 0.0152 | 0.1464 | 0.0226 | 0.5018 | 0.0137 | 76.97 |
| 10 | 0.0150 | 0.1019 | 0.0152 | 0.1480 | 0.0228 | 0.5020 | 0.0138 | 77.05 |
| 11 | 0.0150 | 0.1030 | 0.0153 | 0.1490 | 0.0228 | 0.5059 | 0.0141 | 77.07 |
| 12 | 0.0150 | 0.1030 | 0.0153 | 0.1495 | 0.0230 | 0.5061 | 0.0141 | 77.11 |
| 13 | 0.0150 | 0.1043 | 0.0157 | 0.1530 | 0.0230 | 0.5061 | 0.0148 | |
| 14 | 0.0154 | | 0.0157 | 0.1543 | 0.0243 | 0.5145 | 0.0153 | |
| 15 | | | 0.0159 | | 0.0252 | | | |
| **Mean** | **0.0148** | **0.0996** | **0.0150** | **0.1432** | **0.0223** | **0.4949** | **0.0132** | **76.87** |
| **Std Dev** | 0.0003 | 0.0036 | 0.0005 | 0.0073 | 0.0014 | 0.0130 | 0.0012 | 0.16 |
| **$C_{(95\%)}$** | 0.0002 | 0.0021 | 0.0003 | 0.0042 | 0.0008 | 0.0075 | 0.0007 | 0.10 |

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 | Middlesbrough, England | UKAS accreditation 0239 |
| Sheffield Assay Office | Sheffield, England | UKAS accreditation 0012 |
| 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 |
| Genitest, Inc | Montreal, Canada | PRI accreditation 123077 |
| 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 | |
| Coleshill Laboratories Ltd | Birmingham, England | |
| Analyticka Laborator Lithea sro | Brno, Czech Republic | |

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 | 1, 5, 7, 8, 10-12 | 9 | 2 | gravimetric (cupferron) |
| | | | 3, 4, 6 | volumetric (iodate) |
| Lead | 1-3, 5-8, 10-12 | 9 | 4 | gravimetric (sulFate) |
| Zinc | 1, 4, 6-8, 10-13 | 2 | 3, 5, 9 | volumetric (EDTA |
| Iron | 1, 2, 4, 7-9, 11-13 | 3, 5 | 6 | volumetric (redox) |
| | | | 10 | photometric (o-phenanthroline |
| Nickel | 1, 3, 4, 6, 8, 10, 11 | 9 | 2 | gravimetric (dimethyl glyoxime) |
| | | | 5, 7 | photometric (dimethyl glyoxime) |
| Aluminium | 1, 3, 4, 6-8, 11-14 | 5, 9 | 2 | volumetric (EDTA) |
| | | | 10 | photometric (chrome azurol S) |
| Arsenic | 1-7, 11, 12 | 9 | 8 | photometric (turbidity) |
| | | | 10 | gravimetric (sulfide) |
| Phosphorus | 2, 3, 7, 9-11 | - | 1, 4 | photometric (molybdenum yellow) |
| | | | 5, 6, 8 | volumetric (alkalimetric) |
| Manganese | 1-3, 5-8, 10-14 | 9 | 4 | volumetric (arsenite) |
| Cobalt | 1, 2, 4-10, 12, 13 | 3 | 11 | gravimetric |
| Bismuth | 1, 3-5, 8-11, 13-15 | 6, 7 | 2 | gravimetric |
| | | | 12 | photometric (iodide) |
| Antimony | 1, 3, 5-10, 12-14 | 4 | 2 | photometric (crystal violet) |
| | | | 11 | volumetric (permanganate) |
| Silicon | 1-7, 11, 13, 15 | - | 8, 10, 12, 14 | photometric (molybdenum yellow) |
| | | | 9 | gravimetric (perchloric acid) |
| Silver | 2-4, 6, 8-14 | 1, 5 | 7 | gravimetric |
| Sulfur | 2, 5, 9 | - | | combustion (IR or volumetric detection) |
| Copper | 2, 4, 9, 11 | - | 1, 3, 5-7, 10 | volumetric (thiosulfate) |
| | | | 8, 12 | electrogravimetric |

## 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 to a depth of 12mm. Material to the rear of the disc, to a depth of ~3mm, 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. This certification will therefore expire in July 2037, although we reserve the right to make changes as issue revisions, in the intervening period.

This product is also available in the form of chippings, for the monitoring and calibration of wet analytical techniques.

The manufacture, analysis and certification of this product were supervised by C Eveleigh, PhD, Technical Director, MBH Analytical Ltd.