

# CERTIFICATE OF ANALYSIS

**43X Z4 (batch C)**

## Certified Reference Material Information

Type: ZINC/ALUMINIUM/COPPER ALLOY (CAST)  
Form and Size: Disc ~50mm diameter  
Produced by: MBH Analytical Ltd  
Certified and supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	Pb	Mg	Al	Cd	Fe	Sn
Value <sup>1</sup>	0.0062	0.0480	4.79	0.0033	0.0017	0.0030
Uncertainty <sup>2</sup>	0.0004	0.0013	0.03	0.0001	0.0002	0.0001

Element	Cu	Ni	Mn	Si	Sb	Bi
Value <sup>1</sup>	2.69	0.0258	0.0153	(0.0012)	(0.0023)	0.0113
Uncertainty <sup>2</sup>	0.03	0.0007	0.0005	-	-	0.0004

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

## Definitions

- <sup>1</sup> 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.
- <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 12th December 2013

C Eveleigh

## **Method of Preparation**

This certified reference material was produced from commercial zinc, with the major alloys and traces added as pure elements or master alloys. The metal was cast from the bulk melt by sequential transfer of aliquots into individual iron chill moulds. At least 1mm has been machined from the working face of each disc, to minimise surface effects.

## **Sampling**

Samples for chemical analysis were taken from throughout the casting process. In addition, at least 10% of all discs, chosen at random from the complete batch, were checked for homogeneity.

## **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.

## **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: Zinc and zinc 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.

For OES the sample should be of sufficient mass to prevent excess heating during sparking, and the discharge chamber should be regularly cleaned as directed by the instrument manufacturer.

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	Mg	Al	Cd	Fe	Sn
1	0.0050	0.0456	4.720	0.0031	0.0014	0.0026
2	0.0050	0.0461	4.722	0.0031	0.0015	0.0027
3	0.0052	0.0462	4.755	0.0031	0.0015	0.0027
4	0.0058	0.0472	4.764	0.0032	0.0015	0.0028
5	0.0059	0.0476	4.777	0.0032	0.0016	0.0029
6	0.0060	0.0477	4.783	0.0032	0.0016	0.0030
7	0.0062	0.0480	4.786	0.0033	0.0016	0.0030
8	0.0063	0.0482	4.798	0.0033	0.0017	0.0030
9	0.0063	0.0484	4.803	0.0033	0.0019	0.0030
10	0.0066	0.0485	4.809	0.0033	0.0020	0.0031
11	0.0069	0.0489	4.811	0.0035	0.0020	0.0032
12	0.0070	0.0491	4.828	0.0035	0.0021	0.0032
13	0.0071	0.0492	4.849	0.0036		0.0032
14	0.0073	0.0500				
15		0.0500				
<b>Mean</b>	<b>0.0062</b>	<b>0.0480</b>	<b>4.785</b>	<b>0.0033</b>	<b>0.0017</b>	<b>0.0030</b>
<b>Std Dev</b>	0.0008	0.0013	0.038	0.0002	0.0002	0.0002
<b>C<sub>(95%)</sub></b>	0.0004	0.0007	0.023	0.0001	0.0002	0.0001

Sample	Cu	Ni	Mn	Si	Sb	Bi
1	2.640	0.0240	0.0139	0.0004	0.0012	0.0104
2	2.647	0.0241	0.0141	0.0006	0.0013	0.0104
3	2.656	0.0251	0.0148	0.0009	0.0015	0.0108
4	2.665	0.0252	0.0150	0.0010	0.0017	0.0110
5	2.667	0.0252	0.0151	0.0010	0.0019	0.0110
6	2.684	0.0254	0.0153	0.0013	0.0022	0.0111
7	2.686	0.0256	0.0153	0.0013	0.0023	0.0113
8	2.688	0.0257	0.0154	0.0016	0.0024	0.0113
9	2.694	0.0257	0.0154	0.0017	0.0026	0.0113
10	2.711	0.0259	0.0156	0.0018	0.0026	0.0114
11	2.721	0.0260	0.0158		0.0027	0.0119
12	2.729	0.0265	0.0160		0.0027	0.0121
13	2.732	0.0266	0.0164		0.0028	0.0121
14	2.740	0.0268	0.0165		0.0030	0.0123
15	2.755	0.0269			0.0032	
16		0.0273				
<b>Mean</b>	<b>2.694</b>	<b>0.0258</b>	<b>0.0153</b>	<b>(0.0012)</b>	<b>(0.0023)</b>	<b>0.0113</b>
<b>Std Dev</b>	0.036	0.0009	0.0007	-	-	0.0006
<b>C<sub>(95%)</sub></b>	0.020	0.0005	0.0004	-	-	0.0004

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 Sheffield Assay Office Universal Scientific Laboratory Pty Ltd 492 Genitest, Inc 123077 Luo Yang Copper Institute of Iron & Steel Technology South-West Aluminium Group TCR Engineering Services Ltd Sargam Metals Pvt Ltd Raghavendra SpectroMet Laboratory Institute of Non-Ferrous Metals Tec-Eurolab London & Scandinavian Met Co Coleshill Laboratories Ltd Sun Metals Corp Lithea sro	Middlesbrough, England Sheffield, England Milperra, NSW, Australia  Montreal, Canada  Luo Yang, He Nan, China Shanghai, China Jiulong Puo, Sichuan, China Mumbai, India Chennai, India Bangalore, India Gliwice, Poland Campogalliano, Italy Rotherham, England Birmingham, England Townsville, Qld, Australia Brno, Czech Republic	UKAS accreditation 0239 UKAS accreditation 0012 NATA accreditation  PRI accreditation  CNAL accreditation 0173 CNAL accreditation 0783 CNAL accreditation T007 NABL accreditation 0367 NABL accreditation 0025 NABL accreditation T371 PCA accreditation AB274 ACCREDIA accreditation 52
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Note: to achieve the above accreditation (eg 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, 6-9, 11, 12, 14	2, 4, 5, 10, 13	
Magnesium	1-5, 7, 9, 12-14	6, 8, 10, 11, 15	
Aluminium	2, 4-6, 10-13	7, 9	1, 3 8 volumetric (EDTA) photometric (chrome azurol S)
Cadmium	1, 3, 4, 8-13	2, 5-7	
Iron	1, 3, 6, 7, 9, 11, 12	2, 4, 5, 8, 10	
Tin	1-4, 6, 7, 10, 12, 13	5, 8, 9, 11	
Copper	4-7, 9, 11-13	1-3, 8, 10, 14, 15	
Nickel	1-4, 8-15	5-7, 16	
Manganese	1, 3-5, 9-14	2, 6-8	
Silicon	1, 4, 7, 8, 10	3	2, 5, 6, 9 photometric (molybdenum blue)
Antimony	2, 3, 5-7, 10-15	1, 4, 8, 9	
Bismuth	2, 3, 5-8, 11-14	1, 4, 9, 10	

## 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 unidirectional solidification effects associated with this method of casting may 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 15mm. Material to the rear of the disc, to a depth of ~5mm, is not certified.

There is a possibility for microstructural changes within this sample due to recrystallisation; and diffusion effects may lead to the concentration of some elements at the surface. For X-ray and other superficial sampling techniques, it is therefore recommended that the surface is refreshed immediately prior to use. In all other respects, this sample 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 original analysis. Technical support for this certification will therefore expire in December 2033, although we reserve the right to make changes as issue revisions, in the intervening period.

This sample 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.