

# CERTIFICATE OF ANALYSIS

## 43X Z15 (batch C)

### Certified Reference Material Information

Type: ZINC / ALUMINIUM / COPPER (CAST)  
Form and Size: Disc 50mm Diameter x 20mm Thickness  
Manufactured by: G Rhodes  
Certified and Supplied by: MBH Analytical Limited

### Certified Analysis

#### Percentage element by weight

Element	Pb	Mg	Al	Cd	Fe	Sn	Cu
Value <sup>1</sup>	<b>0.0054</b>	<b>0.0024</b>	<b>7.36</b>	<b>0.0030</b>	<b>0.009</b>	<b>0.004</b>	<b>1.53</b>
Uncertainty <sup>2</sup>	0.0007	0.0004	0.12	0.0001	0.002	0.001	0.02

Element	Ni	Mn	Cr	Ti	Sb	Bi	Si
Value <sup>1</sup>	<b>0.0019</b>	<b>0.0020</b>	<b>0.0025</b>	<b>0.0020</b>	<b>0.005</b>	<b>0.005</b>	<b>(0.011)</b>
Uncertainty <sup>2</sup>	0.0003	0.0002	0.0002	0.0002	0.001	0.001	-

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 22nd January 2004

C Eveleigh

## **Method of Preparation**

This reference material was produced from master alloys and commercial-purity zinc and aluminium. The metal was cast from the bulk melt by sequential transfer of aliquots into individual iron chill moulds. At least 1mm was machined from the upper and lower surfaces of each disc, to minimise surface effects.

## **Sampling**

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

## **Homogeneity**

The discs were checked for sample and batch uniformity using an optical emission spectrometer. One additional disc was checked for vertical uniformity using the same method.

For each of the surfaces checked, the differences between the averaged result and the overall mean value were evaluated to ensure that the overall homogeneity of the material comprising the batch satisfied the definition given in ISO guide 30 - 1992.

Using the meaned data for each surface, standard deviation values were derived for each element. These values were combined with the 95% half-width confidence intervals ( $C_{(95\%)}$ ) obtained from the wet analysis programme, using the square-root of the summed squares, to derive the final uncertainty values.

The vertical uniformity check showed that this material is of satisfactory homogeneity, for all certified elements, for at least the first 15 mm of depth from the original chilled face.

## **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 ISO Guide 25 – 1990, or EN ISO/IEC 17025 - 2000, 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**

Most of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to a national authority. It is part of the 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 primary reference materials.

## **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 four 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	Mg	Al	Cd	Fe	Sn	Cu
1	0.0040	0.0017	7.22	0.0026	0.0065	0.0029	1.51
2	0.0047	0.0019	7.23	0.0027	0.0076	0.003	1.52
3	0.005	0.002	7.32	0.0029	0.008	0.003	1.52
4	0.005	0.002	7.33	0.003	0.0084	0.0031	1.53
5	0.0053	0.0022	7.35	0.0030	0.0084	0.0036	1.53
6	0.0057	0.0025	7.38	0.0030	0.0088	0.004	1.534
7	0.006	0.0026	7.44	0.003	0.010	0.005	1.56
8	0.006	0.0027	7.47	0.0031	0.010	0.0052	1.57
9	0.007	0.003	7.49	0.0032	0.010	0.0053	
10		0.0033		0.0032			
11				0.0033			
<b>Mean</b>	<b>0.0054</b>	<b>0.0024</b>	<b>7.36</b>	<b>0.0030</b>	<b>0.0086</b>	<b>0.0039</b>	<b>1.534</b>
<b>Std Dev</b>	0.0009	0.0005	0.10	0.0002	0.0012	0.0010	0.021
<b>C<sub>(95%)</sub></b>	0.0007	0.0004	0.07	0.0001	0.0009	0.0008	0.017

Sample	Ni	Mn	Cr	Ti	Sb	Bi	Si
1	0.0015	0.0017	0.002	0.0017	0.0047	0.0047	0.005
2	0.0016	0.0018	0.002	0.0018	0.0047	0.0047	0.007
3	0.0017	0.0019	0.0022	0.002	0.005	0.005	0.0079
4	0.0018	0.002	0.0025	0.002	0.005	0.006	0.0086
5	0.0018	0.002	0.0025	0.0021	0.005	0.006	0.01
6	0.002	0.0020	0.0026	0.0021	0.006	0.006	0.0151
7	0.0020	0.0021	0.0026	0.0023			0.016
8	0.002	0.0022	0.0027				0.018
9	0.0024	0.0023	0.0030				
<b>Mean</b>	<b>0.0019</b>	<b>0.0020</b>	<b>0.0025</b>	<b>0.0020</b>	<b>0.0051</b>	<b>0.0054</b>	<b>0.011</b>
<b>Std Dev</b>	0.0003	0.0002	0.0003	0.0002	0.0005	0.0007	0.005
<b>C<sub>(95%)</sub></b>	0.0002	0.0001	0.0002	0.0002	0.0005	0.0007	0.004

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

RoTech Laboratories	Wednesbury, England	UKAS accreditation 0366
LGC-NW Ltd	Runcorn, England	UKAS accreditation 1214
London & Scandinavian Met Co Ltd	Rotherham, England	UKAS accreditation 1091
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Bodycote Materials Testing Ltd	Middlesbrough, England	UKAS accreditation 0239
Metals Technology Testing Ltd	Sheffield, England	UKAS accreditation 0963
Laboratory Testing Inc	Hatfield, PA, USA	A2LA accreditation 0117
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Central Iron & Steel Research Inst	Beijing, China	CNAL accreditation 0435
Coleshill Laboratories Ltd	Coleshill, England	
Shiva Analyticals Ltd	Bangalore, India	

Note: to achieve National Accreditation (eg UKAS, A2LA, NATA, CNAL), 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, 4, 6, 8, 9	3, 5, 7	2 square-wave polarography
Magnesium	2, 4, 7-10	1, 3, 5, 6	
Aluminium	1, 7-9	4, 5	2, 3, 6 volumetric (EDTA)
Cadmium	2, 4, 5, 7-9	1, 6, 10, 11	3 square-wave polarography
Iron	3, 5, 6, 8, 9	1, 4, 7	2 photometric (sulfosalicylic acid)
Tin	2-4, 9	5, 6, 7, 8	1 photometric (phenyl fluorone)
Copper	2, 3, 6, 7	4, 5, 8	1 photometric (BCO)
Nickel	1-3, 7-9	4, 5, 6	
Manganese	1, 2, 4-6, 9	3, 7	8 photometric (periodate)
Chromium	2, 5-7, 9	1, 3, 4, 8	
Titanium	1, 2, 4-6	3, 7	
Antimony	1, 5, 6	2, 3, 4	
Bismuth	2, 3, 6	1, 4, 5	
Silicon	2-4, 7, 8	1, 5, 6	

## Notes

This certificate has been up-issued to include new data, and to improve clarity of presentation. This Certified Reference Material was originally certified in September 2000. It has now been produced and certified in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, taking into account the requirements of ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The combination of alloying elements used in a complex material of this type, coupled with the unidirectional solidification effects associated with semi-chill casting, may lead 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 ~5mm, is not certified.

This material will remain stable 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 certification. This certification will therefore expire in September 2020, 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.