

# CERTIFICATE OF ANALYSIS

## 41X GLV1 (batch D)

### Certified Reference Material Information

Type:	ZINC GALVANIZING 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	Ni
Value <sup>1</sup>	0.0224	0.0008	0.202	0.0100	0.021	0.0019
Uncertainty <sup>2</sup>	0.0006	0.0001	0.003	0.0005	0.002	0.0001

Element	Sn	Cu	Co	Bi	Sb	As
Value <sup>1</sup>	0.0134	0.0169	0.0003	0.0040	0.0010	0.0002
Uncertainty <sup>2</sup>	0.0008	0.0005	0.0001	0.0001	0.0002	0.0001

### 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 23<sup>rd</sup> October 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, targeting the nominal composition indicated for UNS Z81330. 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, about 10% of all discs, chosen at random from the complete batch, were selected for non-destructive homogeneity testing.

## **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	Ni
1	0.0209	0.00066	0.195	0.0095	0.0189	0.0016
2	0.0210	0.00077	0.195	0.0095	0.0196	0.0018
3	0.0217	0.0008	0.197	0.0096	0.0197	0.0018
4	0.0217	0.0008	0.197	0.0097	0.0201	0.0019
5	0.0219	0.0008	0.198	0.0097	0.0201	0.0019
6	0.0220	0.0008	0.199	0.0097	0.0203	0.0019
7	0.0222	0.0008	0.202	0.0100	0.0204	0.0019
8	0.0223	0.0008	0.202	0.0100	0.0204	0.0019
9	0.0224	0.00080	0.203	0.0102	0.0205	0.0019
10	0.0224	0.00082	0.204	0.0102	0.0208	0.0019
11	0.0229	0.0009	0.204	0.0102	0.0211	0.0020
12	0.0230	0.0009	0.205	0.0102	0.0211	0.0020
13	0.0232	0.0009	0.206	0.0102	0.0221	0.0020
14	0.0232	0.00091	0.206	0.0104	0.0223	0.0020
15	0.0235		0.207	0.0104	0.0225	0.0020
16	0.0235		0.209	0.0104		0.0020
17				0.0105		0.0022
<b>Mean</b>	<b>0.0224</b>	<b>0.00082</b>	<b>0.202</b>	<b>0.0100</b>	<b>0.0207</b>	<b>0.0019</b>
<b>Std Dev</b>	0.0008	0.00007	0.004	0.0003	0.0010	0.0001
<b>C<sub>(95%)</sub></b>	0.0004	0.00004	0.002	0.0002	0.0006	0.0001

Sample	Sn	Cu	Co	Bi	Sb	As
1	0.0119	0.0158	0.00020	0.0038	0.00052	0.0001
2	0.0122	0.0160	0.0002	0.0039	0.00076	0.0001
3	0.0124	0.0163	0.0002	0.0039	0.0009	0.00014
4	0.0124	0.0166	0.00026	0.0039	0.0009	0.00014
5	0.0124	0.0166	0.00028	0.0040	0.00092	0.00019
6	0.0128	0.0167	0.00028	0.0040	0.0010	0.0002
7	0.0128	0.0167	0.0003	0.0040	0.0010	0.0002
8	0.0141	0.0168	0.0003	0.0041	0.0010	0.0002
9	0.0145	0.0170	0.0003	0.0041	0.0011	0.00025
10	0.0146	0.0172	0.0003	0.0042	0.00110	0.0003
11	0.0150	0.0172	0.0003	0.0042	0.0014	
12	0.0151	0.0174	0.00031	0.0042		
13		0.0174	0.0004	0.0043		
14		0.0175		0.0043		
15		0.0176				
16		0.0177				
<b>Mean</b>	<b>0.0134</b>	<b>0.0169</b>	<b>0.00028</b>	<b>0.0040</b>	<b>0.00096</b>	<b>0.00018</b>
<b>Std Dev</b>	0.0012	0.0006	0.00006	0.0002	0.00022	0.00006
<b>C<sub>(95%)</sub></b>	0.0008	0.0003	0.00003	0.0001	0.00015	0.00005

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, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 492
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
South-West Aluminium Group	Jiulong Puo, Sichuan, China	CNAL accreditation T007
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Sargam Laboratory Pvt Ltd	Chennai, India	NABL accreditation 0025
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
London & Scandinavian Met Co	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Nyrstar Hobart Pty Ltd	Hobart, Tas, Australia	
Sun Metals Corp	Townsville, Qld, Australia	
Votorantim Metais	Lima, Peru	
Lithea sro	Brno, Czech Republic	

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	FAAS	OTHER
Lead	1-3, 5-7, 9-11, 14-16	4, 8, 12, 13	
Magnesium	1-7, 10, 12-14	8, 9, 11	
Aluminium	1-4, 6-8, 12-16	5, 11	9 photometric (chrome azurol S) 10 volumetric (EDTA)
Cadmium	2-7, 11-17	1, 8-10	
Iron	1-6, 8, 10, 12, 13, 15	7, 9, 11	14 photometric (orthophenanthroline)
Nickel	1, 6-17	2-4	5 volumetric (DMGO)
Tin	1-6, 8, 12	9, 10	7, 11 photometric (phenylfluorone)
Copper	2-7, 9, 10, 13-16	1, 8, 11, 12	
Cobalt	2-4, 6, 9-13	1, 5, 7, 8	
Bismuth	1, 3-5, 7-9, 12-14	2, 11	6, 10 photometric (brucine)
Antimony	1-10	-	11 photometric (iodide)
Arsenic	2-5, 7-9	1, 6	10 ICP-MS

## Notes

This Certified Reference Material has been produced and certified, wherever possible, 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 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, 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 October 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.