

CERTIFICATE OF ANALYSIS

45X ZN-AL3 (batch B) (revision 1)

Reference Material Information

Type: ZINC/ALUMINIUM 'GALVALUME' (CHILL CAST)

Form and Size: Disc 55mm Diameter x ~6mm Thickness
'mushroom' disc with central spigot

Produced by: J Watson

Certified and supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Zn	Si	Fe	Cu	Sn	Pb
Value ¹	45.2	0.606	0.047	0.0088	0.0034	0.0062
Uncertainty ²	0.4	0.013	0.002	0.0003	0.0004	0.0002

Element	Mg	Ti	Ca	Li	Al
Value ¹	(0.00021)	0.020	0.0004	(0.0004)	(54.5)
Uncertainty ²	-	0.001	0.0001	-	-

Definitions

- ¹ 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.
- 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:

MBH ANALYTICAL LIMITED _____

on 15th October 2015

C Eveleigh

Method of Preparation

This reference material was produced from commercial-purity zinc and aluminium, with the traces added as master alloys or pure elements. Discs were cast from the bulk melt by sequential transfer of aliquots into individual iron double-chill moulds.

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 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, 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.
	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	Zn	Si	Fe	Cu	Sn	Pb
1	44.58	0.584	0.0449	0.0080	0.0026	0.0057
2	45.00	0.596	0.0460	0.0080	0.0028	0.0058
3	45.08	0.597	0.0462	0.0084	0.0028	0.0060
4	45.26	0.598	0.0463	0.0087	0.0030	0.0060
5	45.30	0.600	0.0465	0.0088	0.0031	0.0062
6	45.34	0.611	0.0465	0.0089	0.0031	0.0062
7	45.94	0.614	0.0470	0.0089	0.0038	0.0062
8		0.615	0.0470	0.0090	0.0039	0.0063
9		0.640	0.0470	0.0091	0.0040	0.0065
10			0.0486	0.0092	0.0042	0.0065
11			0.0490	0.0095	0.0043	0.0065
12			0.0493			0.0070
Mean	45.2	0.606	0.0470	0.0088	0.0034	0.0062
Std Dev	0.4	0.016	0.0013	0.0005	0.0006	0.0004
C_(95%)	0.4	0.012	0.0008	0.0003	0.0004	0.0002

Sample	Mg	Ti	Ca	Li	Al
1	0.00010	0.0190	0.00030	0.00032	54.19
2	0.00017	0.0190	0.00040	0.00040	54.82
3	0.00018	0.0190	0.00041	0.00041	
4	0.00020	0.0199	0.00041	0.00042	
5	0.00020	0.0200	0.00044	0.00045	
6	0.00020	0.0203	0.00045	<0.0005	
7	0.00021	0.0205	0.00047	<0.0005	
8	0.00022	0.0207		<0.001	
9	0.00030	0.0209			
10	0.00036	0.0220			
Mean	(0.00021)	0.0201	0.00041	0.00040	(54.5)
Std Dev	-	0.0010	0.00006	0.00005	-
C_(95%)	-	0.0007	0.00005	0.00006	-

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

Bodycote Materials Testing	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 Co	Luo Yang, He Nan, China	CNAL accreditation 0173
South-West Aluminium Group	Jiulong Puo, Sichuan, China	CNAL accreditation T007
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation 0371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

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					OTHER
	ICP-AES	ICP-MS	OES	FAAS		
Zinc	3-5, 7	-	6	-	1, 2	volumetric (EDTA)
Silicon	-	-	-	-	1-5, 7, 9	gravimetric (perchloric acid)
Iron	2-4, 9-12	-	5	7, 8	6, 8	photometric (molybdenum blue)
Copper	1-3, 5, 7-10	-	-	4, 6, 11	1	photometric (orthophenanthroline)
Tin	3, 4, 6-8, 10, 11	9	5	1	6	photometric (sulfosalicylic acid)
Lead	1, 3, 5-7, 10-12	-	-	2, 4, 8, 9	2	photometric (phenyl fluorone)
Magnesium	1, 5, 6, 9, 10	3	7	2, 4, 8		
Titanium	2-5, 9, 10	1	-	6	7, 8	photometric (di-antipyryl methane)
Calcium	1-4	5	-	6, 7		
Lithium	1, 2, 6-8	4	-	3, 5		
Aluminium	-	-	1	-	2	gravimetric

Notes

This Reference Material has been produced and certified in accordance with the requirements of ISO Guide 80, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The rapid solidification effects associated with double-chill casting have ensured a workable degree of homogeneity within the bulk of the disc. However, the central portion (adjacent to the spigot) contains segregated material, and sampling from this area should be avoided.

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 final analysis. This certification will therefore expire in October 2035, 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 C Eveleigh, PhD, Technical Director, MBH Analytical Ltd.

This product has been subjected to additional analysis and recertified, after substantial input from various users and other advisory bodies.

This product is not certified as a CRM, and all results arising should be treated with caution.

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