

CERTIFICATE OF ANALYSIS

43X SC4 (batch A)

Certified Reference Material Information

Type: ZINC SPIN-CASTING ALLOY (CAST)
Form and Size: Disc 50mm Diameter x 20mm Thickness
Produced by: MBH Analytical Ltd
Certified and supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Al	Mg	Cu	Fe	Pb	Cd
Value ¹	4.35	0.093	1.122	0.022	0.0064	0.0058
Uncertainty ²	0.03	0.002	0.009	0.002	0.0005	0.0003

Element	Sn	Ni	Cr	Mn	Si
Value ¹	0.0056	0.0249	0.009	0.044	0.022
Uncertainty ²	0.0003	0.0014	0.001	0.002	0.002

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:

MBH ANALYTICAL LIMITED _____

on 2nd March 2010

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	Al	Mg	Cu	Fe	Pb	Cd
1	4.280	0.0885	1.100	0.0185	0.0055	0.0055
2	4.299	0.0891	1.100	0.0189	0.0056	0.0055
3	4.321	0.0896	1.111	0.0201	0.0060	0.0055
4	4.337	0.0900	1.112	0.0204	0.0062	0.0056
5	4.337	0.0921	1.113	0.0205	0.0064	0.0057
6	4.358	0.0925	1.115	0.0205	0.0064	0.0058
7	4.358	0.0925	1.121	0.0208	0.0065	0.0059
8	4.366	0.0929	1.125	0.0209	0.0065	0.0059
9	4.367	0.0943	1.126	0.0211	0.0065	0.0059
10	4.371	0.0946	1.129	0.0223	0.0068	0.0060
11	4.383	0.0958	1.132	0.0224	0.0068	0.0061
12	4.394	0.0967	1.139	0.0250	0.0073	0.0062
13	4.421	0.0975	1.145	0.0255		0.0063
14			1.146	0.0255		
Mean	4.353	0.0928	1.122	0.0216	0.0064	0.0058
Std Dev	0.038	0.0030	0.015	0.0023	0.0005	0.0003
C_(95%)	0.023	0.0018	0.009	0.0013	0.0003	0.0002

Sample	Sn	Ni	Cr	Mn	Si
1	0.0048	0.0231	0.0068	0.0418	0.0185
2	0.0048	0.0235	0.0081	0.0423	0.0191
3	0.0052	0.0240	0.0082	0.0428	0.0206
4	0.0053	0.0241	0.0085	0.0430	0.0218
5	0.0053	0.0243	0.0087	0.0432	0.0225
6	0.0054	0.0249	0.0090	0.0441	0.0227
7	0.0057	0.0251	0.0091	0.0442	0.0235
8	0.0057	0.0254	0.0093	0.0442	0.0238
9	0.0058	0.0255	0.0100	0.0449	0.0240
10	0.0058	0.0271	0.0102	0.0451	0.0243
11	0.0061	0.0273	0.0117	0.0453	0.0252
12	0.0061			0.0454	
13	0.0066			0.0461	
Mean	0.0056	0.0249	0.0091	0.0440	0.0224
Std Dev	0.0005	0.0014	0.0013	0.0013	0.0022
C_(95%)	0.0003	0.0009	0.0008	0.0008	0.0015

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 Materials Testing 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
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
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 Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
Eastern Alloys Inc	Maybrook, NY, USA	
Coleshill Laboratories Ltd	Birmingham, England	
Nyrstar Hobart Pty Ltd	Hobart, Tas, Australia	

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
Aluminium	5, 6, 9, 11, 13	2, 3, 12	1 volumetric (EDTA) 4 gravimetric (8-hydroxyquinoline) 7, 8, 10 photometric (chrome azurol S)
Magnesium	1-6, 10, 12	7-9, 11, 13	
Copper	3, 5, 8-13	1, 2, 4, 6, 7, 14	
Iron	2, 3, 8, 10-13	1, 4, 7, 9, 14	5 photometric (orthophenanthroline) 6 photometric (sulfosalicylic acid)
Lead	1, 3, 5, 6, 9-11	2, 4, 7, 8, 12	
Cadmium	2-4, 8-10, 12, 13	1, 5-7, 11	
Tin	1-3, 5-8, 13	4, 9, 12	10, 11 photometric (phenylfluorone)
Nickel	1, 4, 6, 9, 10	2, 3, 5, 7, 8, 11	
Chromium	1-4, 10, 11	5-9	
Manganese	1, 2, 7-9, 11, 13	3, 4, 6, 10, 12	5 photometric (periodate)
Silicon	2, 4, 5, 9, 11	3	1, 6-8, 10 photometric (molybdenum blue)

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2000, 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 of the disc, to a depth of 13mm. Material to the rear of the disc, to a depth of ~7mm, is not certified.

This material is liable to superficial corrosion, and there is some possibility of microstructural changes due to recrystallisation; however, it will otherwise remain stable indefinitely, provided adequate precautions are taken to protect it from cross-contamination and extremes of temperature. All production records will be retained for a period of 20 years from the date of original analysis. This certification will therefore expire in March 2030, 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.