

CERTIFICATE OF ANALYSIS

41X 0336Zn2 (batch N)

Certified Reference Material Information

Type: ZINC WITH IMPURITIES (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	Cu
Value ¹	0.492	0.0568	1.000	0.147	0.0283	0.0547	0.216
Uncertainty ²	0.005	0.0014	0.013	0.002	0.0013	0.0012	0.003

Element	Co	Mn	Ni	Bi	Sb	Tl	Ag
Value ¹	0.0190	0.0227	0.0047	0.0043	0.0019	0.0016	0.0131
Uncertainty ²	0.0007	0.0003	0.0003	0.0003	0.0002	0.0002	0.0007

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 19th December 2017
C Eveleigh

Method of Preparation

This reference material was produced from commercial-purity 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 lower surface 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 batch, were selected for non-destructive homogeneity testing.

Homogeneity

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. The testing procedure was in accordance with ASTM E826 and the material found acceptable.

From the test data, through-batch variation values were derived for each element as an indicator of any minor compositional variation (as determined for the specific sample size and other limitations of 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 compositional variation of the batch for each element has been quantified by a programme of non-destructive application testing, described 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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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	Cu
1	0.4812	0.0538	0.9743	0.1430	0.0266	0.0523	0.2060
2	0.4830	0.0539	0.9791	0.1440	0.0266	0.0528	0.2093
3	0.4867	0.0540	0.9811	0.1450	0.0268	0.0531	0.2100
4	0.4880	0.0548	0.9839	0.1450	0.0277	0.0533	0.2120
5	0.4880	0.0556	0.9842	0.1461	0.0278	0.0536	0.2130
6	0.4900	0.0557	0.9987	0.1467	0.0281	0.0539	0.2137
7	0.4900	0.0561	1.0020	0.1478	0.0282	0.0544	0.2150
8	0.4906	0.0569	1.0040	0.1480	0.0283	0.0548	0.2173
9	0.4918	0.0572	1.0060	0.1480	0.0286	0.0549	0.2181
10	0.4970	0.0577	1.0110	0.1488	0.0293	0.0557	0.2198
11	0.5003	0.0578	1.0159	0.1498	0.0296	0.0559	0.2200
12	0.5035	0.0585	1.0280	0.1507	0.0300	0.0560	0.2206
13	0.5051	0.0591	1.0370	0.1525	0.0304	0.0566	0.2210
14		0.0597				0.0578	0.2214
15		0.0606					0.2220
Mean	0.4919	0.0568	1.0004	0.1473	0.0283	0.0547	0.2159
Std Dev	0.0074	0.0022	0.0195	0.0027	0.0013	0.0016	0.0051
C_(95%)	0.0045	0.0012	0.0118	0.0017	0.0008	0.0009	0.0028

Sample	Co	Mn	Ni	Bi	Sb	Tl	Ag
1	0.0172	0.0213	0.0044	0.0037	0.0014	0.00130	0.0109
2	0.0173	0.0220	0.0044	0.0038	0.0015	0.00147	0.0111
3	0.0176	0.0224	0.0045	0.0039	0.0017	0.00150	0.0111
4	0.0182	0.0225	0.0046	0.0039	0.0018	0.00150	0.0124
5	0.0184	0.0226	0.0046	0.0040	0.0019	0.00156	0.0126
6	0.0185	0.0226	0.0047	0.0043	0.0020	0.00170	0.0127
7	0.0189	0.0227	0.0047	0.0044	0.0020	0.00170	0.0128
8	0.0190	0.0227	0.0047	0.0046	0.0020	0.00170	0.0136
9	0.0190	0.0228	0.0048	0.0046	0.0020	0.00185	0.0136
10	0.0192	0.0228	0.0048	0.0047	0.0023	0.00212	0.0139
11	0.0199	0.0228	0.0048	0.0049			0.0140
12	0.0199	0.0231	0.0050				0.0140
13	0.0201	0.0232	0.0050				0.0140
14	0.0208	0.0232					0.0141
15	0.0212	0.0236					0.0153
Mean	0.0190	0.0227	0.0047	0.0043	0.0019	0.00164	0.0131
Std Dev	0.0012	0.0005	0.0002	0.0004	0.0003	0.00023	0.0013
C_(95%)	0.0007	0.0003	0.0001	0.0003	0.0002	0.00016	0.0007

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 Ltd
Sheffield Analytical Services
Universal Scientific Laboratory Pty Ltd
Genitest, Inc
Shanghai Jinyi Test Tech Co
Shandong Metallurgical & Science Research
Luo Yang Copper
Raghavendra SpectroMet Laboratory
TCR Engineering Services Ltd
Institute of Non-Ferrous Metals
Tec-Eurolab
Mineral & Metallurgical Laboratories
INCDMNR-IMNR
AMG Superalloys UK Ltd
Coleshill Laboratories Ltd
Analyticka Laborator Lithea sro

Middlesbrough, England
Sheffield, England
Milperra, NSW, Australia
Montreal, Canada
Shanghai, China
Jinan, Shandong, China
Luo Yang, He Nan, China
Bangalore, India
Mumbai, India
Gliwice, Poland
Campogalliano, Italy
Bangalore, India
Pantelimon, Romania
Rotherham, England
Coleshill, England
Brno, Czech Republic

UKAS accreditation 0239
UKAS accreditation 0012
NATA accreditation 0492
PJ accreditation L17-153
CNAS accreditation L0041
CNAS accreditation 1461
CNAL accreditation 0173
NABL accreditation T371
NABL accreditation 0367
PCA accreditation AB274
ACCREDIA accreditation 52

Note: to achieve the above accreditation (UKAS, 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, 7-11, 13	4, 6, 12	5 gravimetric (sulfate)
Magnesium	2-5, 8-10, 12-15	6, 7, 11	1 gravimetric (pyrophosphate)
Aluminium	1-3, 5, 6, 9, 11-13	4, 7, 8	10 volumetric (EDTA)
Cadmium	1, 3-5, 7, 10-13	6, 8, 9	2 gravimetric (molybdate)
Iron	2, 5-8, 10, 11, 13	3, 4, 9, 12	1 photometric (orthophenanthroline)
Tin	1, 2, 5-7, 9, 10, 12-14	3, 8, 11	4 volumetric (iodate)
Copper	1, 2, 4, 6-10, 12-14	3, 5, 11	15 electrogravimetric
Cobalt	1, 3-8, 10-12, 15	2, 9, 14	13 gravimetric
Manganese	1-4, 8-10, 13-15	5, 6, 11, 12	7 volumetric (arsenite)
Nickel	3, 5-12	1, 2, 4, 13	
Bismuth	1-4, 6-8, 10, 11	5, 9	
Antimony	1, 3-9	2, 10	
Thallium	1-4, 7-10	5, 6	
Silver	1, 3-5, 8-10, 12-15	2, 6, 7, 11	

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, 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 14mm. Material to the rear of the disc, to a depth of ~6mm, is not certified.

This material is liable to superficial corrosion. There is also a possibility for microstructural changes 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 this certificate. Technical support for this certification will therefore expire in December 2037, 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.