

# CERTIFICATE OF ANALYSIS

**65X MGB3 (batch C)**

## Certified Reference Material Information

Type: MAGNESIUM / ALUMINIUM / ZINC (CAST)

Form and Size: Disc 40-50mm diameter

Produced by: KRR Metals Ltd

Certified and supplied by: MBH Analytical Ltd

## Assigned Values

Element	Al	Zn	Mn	Cu	Si	Fe	Ni
Value <sup>1</sup>	3.38	0.711	0.277	0.0222	0.042	0.0028	0.0027
Uncertainty <sup>2</sup>	0.03	0.010	0.010	0.0012	0.003	0.0004	0.0001

Element	Be	Ca	Sn	Pb	Ag	Cd
Value <sup>1</sup>	0.0019	(0.0033)	0.0017	0.0023	0.0028	0.0025
Uncertainty <sup>2</sup>	0.0002	-	0.0003	0.0002	0.0002	0.0001

Note: values given in parentheses are not certified - they are provided for information only.

## Definitions

- <sup>1</sup> The certified values are derived from the 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 (page 3). When appropriate, these values have been modified to account for additional information from the material homogeneity checks.

## Certified by:

MBH ANALYTICAL LIMITED \_\_\_\_\_

on 24th January 2014

C Eveleigh

## **Method of Preparation**

This reference material was produced from commercial-purity magnesium, with the addition of major alloying ingredients and traces as pure elements or binaries, targeting a composition to match grade AZ31D. All discs are the product of one melt, which was cleaned under a low-melting flux and sequentially cast into iron chill moulds. The first 2mm has been removed from the working face of the discs, to minimise surface effects.

## **Sampling**

Samples for chemical analysis were taken from several positions throughout the casting process. Approximately 10% of all discs were selected for non-destructive homogeneity checking.

## **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:	Magnesium and magnesium alloys are generally prepared by milling or turning on a lathe, avoiding the use of lubricants and ensuring that 'sparking' does not occur during the process. 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, and should be done immediately prior to analysis to minimise the effects of surface oxidation.  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.

## **Safety**

Finely-divided magnesium may ignite. Sand should be available in the event of a fire. Water should never be used.

In OES the sample should be of sufficient mass to prevent excessive heating during sparking and the discharge chamber should be regularly cleaned as directed by the instrument manufacturer.

## Analytical Data

### Percentage element by weight

Sample	Al	Zn	Mn	Cu	Si	Fe	Ni
1	3.337	0.700	0.264	0.0202	0.0368	0.0021	0.0024
2	3.349	0.701	0.267	0.0204	0.0375	0.0025	0.0024
3	3.350	0.701	0.272	0.0204	0.0392	0.0025	0.0025
4	3.364	0.701	0.274	0.0214	0.0404	0.0026	0.0026
5	3.372	0.707	0.275	0.0216	0.0407	0.0028	0.0026
6	3.381	0.708	0.275	0.0220	0.0435	0.0029	0.0026
7	3.382	0.708	0.281	0.0222	0.0441	0.0029	0.0026
8	3.386	0.708	0.281	0.0224	0.0443	0.0030	0.0026
9	3.389	0.720	0.284	0.0225	0.0473	0.0035	0.0027
10	3.399	0.722	0.284	0.0226	0.0482		0.0028
11	3.403	0.727	0.289	0.0229			0.0030
12	3.405	0.729		0.0229			0.0031
13	3.414			0.0243			0.0031
14	3.419			0.0246			
<b>Mean</b>	<b>3.382</b>	<b>0.711</b>	<b>0.277</b>	<b>0.0222</b>	<b>0.042</b>	<b>0.0028</b>	<b>0.0027</b>
<b>Std Dev</b>	0.025	0.011	0.008	0.0013	0.004	0.0004	0.0002
<b>C<sub>(95%)</sub></b>	0.015	0.007	0.005	0.0008	0.003	0.0003	0.0001

Sample	Be	Ca	Sn	Pb	Ag	Cd
1	0.0016	0.0011	0.0012	0.0020	0.0024	0.0022
2	0.00164	0.0015	0.0013	0.0020	0.0026	0.0023
3	0.0017	0.0017	0.0014	0.0021	0.0027	0.0023
4	0.0017	0.0023	0.0015	0.0022	0.0027	0.0024
5	0.0017	0.0027	0.0016	0.0023	0.0027	0.0024
6	0.00186	0.0028	0.0018	0.0023	0.0027	0.0024
7	0.00189	0.0038	0.0020	0.0024	0.0028	0.0024
8	0.00192	0.0042	0.0021	0.0025	0.0028	0.0025
9	0.0020	0.0042	0.0021	0.0025	0.0028	0.0025
10	0.0021	0.0043	0.0022	0.0026	0.0028	0.0026
11	0.0021	0.0046			0.0030	0.0026
12	0.0021	0.0047			0.0031	0.0026
13	0.0021					0.0026
<b>Mean</b>	<b>0.00188</b>	<b>0.0033</b>	<b>0.0017</b>	<b>0.0023</b>	<b>0.0028</b>	<b>0.0025</b>
<b>Std Dev</b>	0.00019	0.0013	0.0004	0.0002	0.0002	0.0001
<b>C<sub>(95%)</sub></b>	0.00012	0.0008	0.0003	0.0002	0.0001	0.0001

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	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
South-West Aluminium Group	Jiulong Puo, Sichuan, China	CNAL accreditation T007
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Bureau Veritas CPS Pvt Ltd	Chennai, India	NABL accreditation 0025
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Coleshill Laboratories Ltd	Birmingham, England	
London & Scandinavian Met Co	Rotherham, England	
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		
	ICP-AES	FAAS	OTHER
Aluminium	1-3, 6, 7, 10, 12-14	4, 8, 9, 11	5 photometric (chrome azurol S)
Zinc	1-3, 7-10, 12	4-6, 11	
Manganese	3, 4, 6, 8-11	1, 5, 7	2 photometric (periodate)
Copper	1-3, 5, 6, 9-11, 14	4, 7, 8, 13	12 photometric (BCO)
Silicon	1, 2, 4, 6, 8, 9	-	3, 5, 7, 10 photometric (molybdenum blue)
Iron	3-5, 7	1, 2, 6, 8	9 photometric (orthophenanthroline)
Nickel	1, 2, 7-12	3-6, 13	
Beryllium	1, 3, 4, 6, 8-11	5, 7, 13	2 photometric (eriochrome cyanine R)
			12 ICP-MS
Calcium	1, 3, 6-9, 12	2, 4, 5, 10, 11	
Tin	2-5, 7, 9, 10	6, 8	1 photometric (phenyl fluorone)
Lead	2-5, 7, 8	1, 6, 9, 10	
Silver	1, 2, 4-6, 9, 10, 12	3, 7, 8, 11	
Cadmium	1-3, 7-9, 11-13	4-6, 10	

## 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. However, testing has shown that the above certification is applicable from the front face of the disc to a depth of about 12mm. Material to the rear of the disc, to a depth of ~3mm (if 15mm diameter) or ~8mm (if 50mm diameter) is not certified.

Precautions should be taken to protect this material from extremes of temperature and atmospheric moisture. It is not chemically stable, and will quickly develop a non-metallic surface film under normal storage conditions. However, it will otherwise remain suitable for long-term use. 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 January 2034, although we reserve the right to make further changes to the certificate, as issue revisions, in the intervening period.

It should be noted that some elements (such as Fe and Si) are not fully-soluble in the matrix. Spectrometer response may not be reliable for such elements.

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.