

# CERTIFICATE OF ANALYSIS

## 64X MGQ9 (batch A)

### Certified Reference Material Information

Type:	MAGNESIUM AS21B-TYPE (CAST)
Form and Size:	Disc 40-50mm diameter x 15-20mm thickness
Produced by:	KRR Metals Ltd
Certified and supplied by:	MBH Analytical Ltd

### Assigned Values

#### Percentage element by weight

Element	Al	Zn	Mn	Cu	Si	Fe	Ni
Value <sup>1</sup>	2.14	0.243	0.068	0.0104	(0.37)	0.0069	0.0020
Uncertainty <sup>2</sup>	0.03	0.004	0.002	0.0005	-	0.0007	0.0002

Element	Sn	Pb	Be	Ce	La	Nd
Value <sup>1</sup>	0.0076	0.0096	0.0015	0.111	0.083	0.114
Uncertainty <sup>2</sup>	0.0006	0.0006	0.0001	0.005	0.004	0.003

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 15<sup>th</sup> May 2010

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. 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.

For 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	2.051	0.230	0.0651	0.0091	0.311	0.0056	0.0015
2	2.085	0.234	0.0653	0.0100	0.319	0.0062	0.0016
3	2.126	0.239	0.0675	0.0100	0.324	0.0062	0.0016
4	2.130	0.240	0.0678	0.0102	0.356	0.0065	0.0018
5	2.140	0.242	0.0682	0.0104	0.358	0.0066	0.0018
6	2.143	0.243	0.0686	0.0104	0.359	0.0066	0.0020
7	2.150	0.244	0.0688	0.0104	0.382	0.0067	0.0021
8	2.152	0.245	0.0700	0.0105	0.411	0.0069	0.0021
9	2.163	0.245	0.0701	0.0105	0.425	0.0072	0.0021
10	2.181	0.245	0.0701	0.0106	0.425	0.0077	0.0022
11	2.212	0.250	0.0705	0.0107		0.0081	0.0023
12		0.250		0.0114		0.0085	0.0023
13		0.251					
<b>Mean</b>	<b>2.14</b>	<b>0.243</b>	<b>0.0684</b>	<b>0.0104</b>	<b>(0.37)</b>	<b>0.0069</b>	<b>0.0020</b>
<b>Std Dev</b>	0.04	0.006	0.0019	0.0005	-	0.0008	0.0003
<b>C<sub>(95%)</sub></b>	0.03	0.004	0.0013	0.0003	-	0.0005	0.0002

Sample	Sn	Pb	Be	Ce	La	Nd
1	0.0062	0.0082	0.0012	0.103	0.0760	0.110
2	0.0064	0.0084	0.0013	0.103	0.0782	0.112
3	0.0064	0.0088	0.0014	0.106	0.0786	0.113
4	0.0066	0.0092	0.0014	0.107	0.0790	0.113
5	0.0076	0.0093	0.0014	0.107	0.0822	0.114
6	0.0077	0.0094	0.0015	0.110	0.0830	0.114
7	0.0077	0.0095	0.0015	0.110	0.0832	0.116
8	0.0080	0.0098	0.0015	0.116	0.0847	0.117
9	0.0084	0.0099	0.0016	0.116	0.0847	0.120
10	0.0084	0.0104	0.0016	0.118	0.0849	
11	0.0085	0.0105	0.0017	0.121	0.0870	
12	0.0086	0.0105	0.0018		0.0892	
13	0.0087	0.0109	0.0018			
<b>Mean</b>	<b>0.0076</b>	<b>0.0096</b>	<b>0.0015</b>	<b>0.111</b>	<b>0.083</b>	<b>0.114</b>
<b>Std Dev</b>	0.0009	0.0008	0.0002	0.006	0.004	0.003
<b>C<sub>(95%)</sub></b>	0.0006	0.0005	0.0001	0.004	0.002	0.002

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 Assay Office  
Universal Scientific Laboratory Pty Ltd  
Laboratory Testing Inc  
Genitest, Inc  
Luo Yang Copper  
Institute of Iron & Steel Technology  
South-West Aluminium Group  
Sargam Metals Pvt Ltd  
Raghavendra Spectrometallurgical Lab  
TCR Engineering Services Pvt Ltd  
US Magnesium LLC  
Coleshill Laboratories Ltd  
Dead Sea Magnesium Research Institute

Middlesbrough, England  
Sheffield, England  
Milperra, NSW, Australia  
Hatfield, PA, USA  
Montreal, Canada  
Luo Yang, He Nan, China  
Shanghai, China  
Jiulong Puo, Sichuan, China  
Chennai, India  
Bangalore, India  
Mumbai, India  
Salt Lake City, UT, USA  
Coleshill, England  
Beer-Sheva, Israel

UKAS accreditation 0239  
UKAS accreditation 0012  
NATA accreditation 0492  
A2LA accreditation 0117  
PRI accreditation 123077  
CNAL accreditation 0173  
CNAL accreditation 0783  
CNAL accreditation T007  
NABL accreditation 0025

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
Aluminium	2-5, 8, 9, 11	6, 10	7 1 volumetric (EDTA) photometric (chrome azurol S)
Zinc	1-3, 6, 9-11, 13	4, 5, 7, 8, 12	7 photometric (periodate)
Manganese	2-5, 8, 10, 11	1, 6, 9	4 photometric (neocuprone)
Copper	1, 3, 8-12	2, 5-7	3, 7, 8 2, 9, 10 photometric (molybdenum blue)
Silicon	1, 4, 5	6	4, 7 gravimetric (perchloric acid) photometric (orthophenanthroline)
Iron	1, 5, 6, 8, 10-12	2, 3, 9	7, 13 photometric (phenyl fluorone)
Nickel	1, 3, 6, 8-12	2, 4, 5, 7	10, 12 photometric (eriochrome cyanine R)
Tin	1-6, 10-12	8, 9	4 photometric (arsenazo III)
Lead	1, 3-5, 9-11, 13	2, 6-8, 12	8 ICP-MS
Beryllium	1-3, 5, 7-9, 11	4, 6, 13	6 photometric (arsenazo III)
Cerium	1-3, 5-7, 9-11	-	10 ICP-MS
Lanthanum	1-5, 7-9, 11, 12	-	4 ICP-MS
Neodymium	1-3, 5-9	-	

## Notes

This Certified Reference Material has been produced 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 (8mm for 50mm diameter discs), 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 stable indefinitely, and is suitable for long-term use.

All production records will be retained for a period of 20 years from the date of this certificate. The technical support for this certification will therefore expire in May 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.