

65X MGA16 A Page 1 of 4 August 2007

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# CERTIFICATE OF ANALYSIS

## 65X MGA16 (batch A)

## **Certified Reference Material Information**

Type: MAGNESIUM / ALUMINIUM / ZINC (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	Ca
Value <sup>1</sup>	6.78	4.03	0.271	0.099	0.023	0.0073	0.0057	0.0024
Uncertainty <sup>2</sup>	0.08	0.06	0.007	0.004	0.002	0.0008	0.0004	0.0002

Element	Sn	Pb	Be	Ag	Cd	Се	La	Hg
Value <sup>1</sup>	0.028	0.050	0.0011	0.0035	0.0066	0.0017	0.0012	0.005
Uncertainty <sup>2</sup>	0.002	0.003	0.0001	0.0002	0.0002	0.0003	0.0003	0.001

## **Definitions**

- The certified values are derived from the results of an interlaboratory testing programme, detailed on page 3.
- 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 29th August 2007

Registered in England, No 1875653 • Registered Office: Holland House, Queens Road, Barnet, EN5 4DJ

#### **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. One additional disc was tested to assess the vertical uniformity of the product. 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 - 2000, 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**

Most of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to a national authority. It is part of the 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 primary reference materials.

#### **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	Ca
1	6.67	3.900	0.248	0.0909	0.0190	0.0063	0.0046	0.0017
2	6.683	3.908	0.256	0.0929	0.0193	0.0064	0.0049	0.0022
3	6.699	3.969	0.260	0.0941	0.0200	0.0067	0.0053	0.0022
4	6.75	4.030	0.268	0.0960	0.0200	0.0068	0.0054	0.0023
5	6.784	4.05	0.270	0.0970	0.0211	0.0071	0.0057	0.0025
6	6.808	4.057	0.271	0.100	0.0223	0.0074	0.0058	0.0025
7	6.83	4.069	0.272	0.102	0.023	0.0074	0.0058	0.0025
8	6.845	4.08	0.278	0.1021	0.0240	0.0075	0.0060	0.0026
9	6.85	4.089	0.278	0.1029	0.025	0.0077	0.0061	0.0028
10	6.865	4.110	0.281	0.1045	0.0265	0.0080	0.0062	
11			0.281 0.285	0.1048 0.105	0.0272	0.0088	0.0064	
12								
Mean	6.778	4.026	0.271	0.0994	0.0225	0.0073	0.0057	0.0024
Std Dev	0.073	0.075	0.011	0.0050	0.0029	0.0007	0.0006	0.0003
C <sub>(95%)</sub>	0.053	0.054	0.007	0.0032	0.0019	0.0005	0.0004	0.0002
	_		_	<b>A</b> -	0.4	0-	Lo	l la
Sample	Sn	Pb	Be	Ag	Cd	Ce	La	Hg
Sample 1	<b>Sn</b> 0.0233	<b>Pb</b> 0.0440	<b>Be</b> 0.0009	<b>Ag</b> 0.0029	0.0060	0.0012	0.0006	<b>пg</b> 0.0039
1				_				_
_	0.0233	0.0440	0.0009	0.0029	0.0060	0.0012	0.0006	0.0039
1 2	0.0233 0.0248	0.0440 0.0470	0.0009 0.0010	0.0029 0.0031	0.0060 0.0060	0.0012 0.0013	0.0006 0.0010	0.0039 0.0042
1 2 3 4 5	0.0233 0.0248 0.0260 0.0264 0.028	0.0440 0.0470 0.0471 0.0472 0.0481	0.0009 0.0010 0.0010 0.0010 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034	0.0060 0.0060 0.0062 0.0063 0.0064	0.0012 0.0013 0.0015 0.0015 0.0015	0.0006 0.0010 0.0011 0.0011 0.0012	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6	0.0233 0.0248 0.0260 0.0264 0.028 0.0285	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493	0.0009 0.0010 0.0010 0.0010 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014	0.0039 0.0042 0.0046 0.0054
1 2 3 4 5 6 7	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515	0.0009 0.0010 0.0010 0.0010 0.0011 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034 0.0035	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516	0.0009 0.0010 0.0010 0.0010 0.0011 0.0011 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034 0.0035 0.0036	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516	0.0009 0.0010 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034 0.0035 0.0036 0.0037	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0067	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8 9	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293 0.0298	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516 0.0536	0.0009 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011 0.0011 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034 0.0035 0.0036 0.0037	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0067 0.0068	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8 9 10	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516 0.0536 0.0537	0.0009 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011 0.0011 0.0012 0.0014	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034 0.0035 0.0036 0.0037 0.0037	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0067 0.0068 0.0068	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8 9 10 11 12	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293 0.0298	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516 0.0536	0.0009 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011 0.0011 0.0011	0.0029 0.0031 0.0031 0.0033 0.0034 0.0035 0.0036 0.0037 0.0037 0.0040 0.0040	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0067 0.0068 0.0068 0.0069	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8 9 10	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293 0.0298	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516 0.0536 0.0537	0.0009 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011 0.0011 0.0012 0.0014	0.0029 0.0031 0.0031 0.0033 0.0034 0.0034 0.0035 0.0036 0.0037 0.0037	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0067 0.0068 0.0068	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8 9 10 11 12	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293 0.0298	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516 0.0536 0.0537	0.0009 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011 0.0011 0.0012 0.0014	0.0029 0.0031 0.0031 0.0033 0.0034 0.0035 0.0036 0.0037 0.0037 0.0040 0.0040	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0067 0.0068 0.0068 0.0069	0.0012 0.0013 0.0015 0.0015 0.0015 0.0020 0.0020 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015	0.0039 0.0042 0.0046 0.0054 0.0060
1 2 3 4 5 6 7 8 9 10 11 12 13	0.0233 0.0248 0.0260 0.0264 0.028 0.0285 0.0288 0.029 0.0293 0.0298 0.0308	0.0440 0.0470 0.0471 0.0472 0.0481 0.0493 0.0515 0.0516 0.0536 0.0537 0.0556	0.0009 0.0010 0.0010 0.0011 0.0011 0.0011 0.0011 0.0011 0.0012 0.0014	0.0029 0.0031 0.0031 0.0033 0.0034 0.0035 0.0035 0.0037 0.0037 0.0040 0.0040	0.0060 0.0060 0.0062 0.0063 0.0064 0.0065 0.0067 0.0068 0.0068 0.0069 0.0069	0.0012 0.0013 0.0015 0.0015 0.0020 0.0020 0.0023 0.0023	0.0006 0.0010 0.0011 0.0011 0.0012 0.0014 0.0015 0.0016	0.0039 0.0042 0.0046 0.0054 0.0060 0.0064

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 Ltd
Sheffield Assay Office
Universal Scientific Laboratory Pty Ltd
Laboratory Testing Inc
Luo Yang Copper
Institute of Iron & Steel Technology
South-West Aluminium Group
Laboratory TUV Nord-Czech
Sargam Metals Pvt Ltd
US Magnesium LLC
Genitest Inc
Coleshill Laboratories Ltd
Dead Sea Magnesium Research Institute

Middlesbrough, England Sheffield, England Milperra, NSW, Australia Hatfield, PA, USA Luo Yang, He Nan, China Shanghai, China Jiulong Puo, Sichuan, China Brno, Czech Republic Chennai, India Salt Lake City, UT, USA Montreal, Canada Coleshill, England Beer-Sheva, Israel UKAS accreditation 0239 UKAS accreditation 0012 NATA accreditation 0492 A2LA accreditation 0117 CNAL accreditation 0773 CNAL accreditation 0783 CNAL accreditation T007 CAI accreditation 1060 NABL accreditation 0025

Note: to achieve National Accreditation (eg UKAS, NATA, A2LA, CNAL, CAI, NABL), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

## **Analytical Methods Used**

ELEMENT	RESULT No. & METHOD					
	ICP-AES	ICP-MS	FAAS		OTHER	
Aluminium	1-3, 5-8	-	10	4, 9	volumetric (EDTA)	
Zinc	1, 3, 4, 6, 7, 10	-	5, 8, 9	2	volumetric (EDTA)	
Manganese	1, 2, 6-8, 10-12	-	3, 4	5, 9	photometric (periodate)	
Copper	1, 3, 4, 7-9, 11, 12	-	2, 5, 6, 10			
Silicon	1-3, 5-7, 9	-	-	4, 8, 10, 11	photometric (molybdenum blue)	
Iron	2-6, 9, 10	-	1, 8, 11	7	photometric (orthophenanthroline)	
Nickel	1, 3-5, 7, 8, 10	11	2, 6	9	volumetric (dimethyl glyoxime)	
Calcium	1, 3, 5, 6, 9	-	2, 4, 7, 8			
Tin	1-5, 7, 10, 11	-	8, 9	6	photometric (phenyl fluorone)	
Lead	1-3, 7, 9-12	-	4-6, 8			
Beryllium	1-3, 5-9, 11	12	4	10	photometric (eriochrome cyanine R)	
Silver	1-4, 9-13	7	5, 6, 8			
Cadmium	1-3, 7-9, 11-13	6	4, 5, 10			
Cerium	1-7, 9	8	-			
Lanthanum	1-7	8	-			
Mercury	2, 4-6	-	-	1	conductimetric (gold film CVD)	
				3	CV-AAS	

## **Notes**

This Certified Reference Material has been produced in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, 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 ~6mm, 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. This certification will therefore expire in August 2027, although we reserve the right to make further 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.