

CERTIFICATE OF ANALYSIS

61X MGP2 (batch A)

Certified Reference Material Information

Type: MAGNESIUM WITH IMPURITIES (CAST)
Form and Size: Disc 45mm Diameter x 20mm Thickness
Produced by: KRR Metals Ltd
Certified and supplied by: MBH Analytical Ltd

Certified Analysis

Percentage element by weight

Element	Al	Zn	Mn	Zr	Cu	Si	Fe	Ni
Value ¹	0.065	0.0122	0.0118	(0.0007)	0.0109	0.029	0.0061	0.0029
Uncertainty ²	0.004	0.0009	0.0008	-	0.0004	0.003	0.0011	0.0002

Element	Ca	Sn	Pb	Ag	Be	Cd	Ce	La
Value ¹	0.0139	0.0073	0.0061	0.003	<0.0001	0.0063	0.0019	0.0014
Uncertainty ²	0.0006	0.0006	0.0007	0.001	-	0.0007	0.0002	0.0003

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

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 4th September 2002

Method of Preparation

This reference material was produced from commercial-purity magnesium, with the addition of traces, mostly as pure elements or binaries. All discs are the product of one melt, which was cleaned under a low-melting flux and poured into cast iron stick moulds. Each stick was sliced to yield a number of discs

Sampling

Samples for chemical analysis and discs for homogeneity checks were taken from several positions throughout the casting process.

Homogeneity

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Multiple measurements were taken from each surface under test.

For each of the surfaces checked, the differences between the averaged result and the overall mean value were evaluated to ensure that the overall homogeneity of the material comprising the batch satisfied the definition given in ISO guide 30 - 1992.

Using the individual data from each check, standard deviation values were derived for each element. These values were combined with the 95% half-width confidence intervals ($C_{(95\%)}$) obtained from the wet analysis programme, using the square-root of the summed squares, to derive the final uncertainty values.

Chemical Analysis

Analysis was carried out on millings taken from samples representative of the product. It was performed by participating laboratories mostly operating within the terms of ISO Guide 25-1990, or the equivalent Guide ISO/IEC 17025-2000, using documented standard methods of analysis.

The individual values listed overpage are the average of each analyst's results.

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 optimise precision and accuracy. 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. Machining tools should be kept sharp to ensure the frictional heat at the tip does not ignite the chips produced. 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

Sample	<u>Percentage element by weight</u>							
	Al	Zn	Mn	Zr	Cu	Si	Fe	Ni
1	0.057	0.010	0.010	0.0002	0.0102	0.024	0.0039	0.0025
2	0.060	0.011	0.0106	0.0006	0.0105	0.0248	0.0044	0.0027
3	0.0645	0.011	0.011	0.0006	0.0106	0.028	0.0056	0.0029
4	0.0648	0.012	0.0114	0.0009	0.0108	0.03	0.0057	0.0029
5	0.065	0.0122	0.0115	0.001	0.0110	0.030	0.0058	0.0030
6	0.0652	0.0124	0.0116	<0.01	0.011	0.030	0.0063	0.003
7	0.066	0.013	0.0124		0.011	0.0312	0.007	0.003
8	0.0677	0.013	0.013		0.011	0.034	0.008	0.003
9	0.0732	0.0132	0.013		0.012		0.008	0.0031
10		0.014	0.013					
Mean	0.0648	0.0122	0.0118	(0.0007)	0.0109	0.029	0.0061	0.0029
Std Dev	0.0045	0.0012	0.0011	-	0.0005	0.003	0.0014	0.0002
C (95%)	0.0035	0.0009	0.0008	-	0.0004	0.003	0.0011	0.0002

Sample	Ca	Sn	Pb	Ag	Be	Cd	Ce	La
1	0.013	0.006	0.0043	0.002	<0.0001	0.0050	0.0017	0.001
2	0.013	0.006	0.0051	0.0022	<0.0001	0.0055	0.0017	0.0013
3	0.0131	0.0067	0.0054	0.0030	<0.0001	0.0055	0.0019	0.0013
4	0.0136	0.0071	0.0054	0.003	<0.0001	0.0064	0.002	0.0014
5	0.0139	0.0075	0.006	0.003	<0.0001	0.0069	0.002	0.0015
6	0.014	0.0077	0.0065	0.0038	0.0001	0.007	0.0022	0.002
7	0.014	0.008	0.0066	0.0039	<0.0002	0.007		
8	0.0146	0.0080	0.007	0.0045		0.007		
9	0.015	0.008	0.007					
10	0.015	0.0084	0.0075					
Mean	0.0139	0.0073	0.0061	0.0032	<0.0001	0.0063	0.0019	0.0014
Std Dev	0.0008	0.0009	0.0010	0.0009	-	0.0008	0.0002	0.0003
C (95%)	0.0005	0.0006	0.0007	0.0007	-	0.0007	0.0002	0.0003

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	Middlesbrough, England	NAMAS accreditation 0239
Birmingham Assay Office	Birmingham, England	NAMAS accreditation 0667
RoTech Laboratories	Wednesbury, England	NAMAS accreditation 0366
Metals Technology Testing Ltd	Sheffield, England	NAMAS accreditation 0963
Sheffield Assay Office	Sheffield, England	NAMAS accreditation 0012
Laboratory Testing Inc	Hatfield, PA, USA	A2LA accreditation 0117
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Central Iron & Steel Research Inst	Beijing, China	CNACL accreditation 0435
Shiva Technologies	Syracuse, NY, USA	
DSM Magnesium Research Institute	Beer-Sheva, Israel	
Shiva Analytical Ltd	Bangalore, India	

Note: to achieve National Accreditation (eg NAMAS, NATA, A2LA, CNACL), test houses were originally required to demonstrate conformity to the general requirements of ISO Guide 25-1990. Since the date of first certification for this product, the Guide has been revised to ISO/IEC 17025.

Analytical Methods Used

ELEMENT	RESULT No. & METHOD			
	ICP-AES	GD-MS	FAAS	OTHER
Aluminium	3, 5, 6, 7, 8	1	2, 4	9 photometric (chromazurol-S)
Zinc	3, 6, 8, 9, 10	2	1, 4, 5, 7	
Manganese	1, 3, 4, 5	8	2, 6, 9, 10	7 photometric (periodate)
Zirconium	1, 2, 3, 5	-	6	4 photometric (arsenazo-III)
Copper	1, 3, 5, 6, 7	-	2, 4, 8, 9	
Silicon	1, 2, 5	8	3, 4, 6	7 photometric (molybdenum blue)
Iron	1, 2, 3, 7, 9	4	5, 8	6 photometric (1,10-phenanthroline)
Nickel	1, 4, 5, 6, 7	2	8, 9	3 photometric (α -fural dioxime)
Calcium	1, 3, 4, 7, 8	6	2, 5, 9, 10	
Tin	2, 3, 4, 6, 7	5	1, 8, 9	10 photometric (phenyl fluorone)
Lead	2, 3, 5, 6	4	7, 8, 9, 10	1 square-wave polarographic
Silver	1, 2, 3, 7	8	4, 5	6 photometric (dithizone)
Beryllium	2, 3, 6, 7	1	4, 5	
Cadmium	3, 4, 5, 6, 7	2	1, 8	
Cerium	1, 2, 3, 4, 5, 6	-	-	
Lanthanum	1, 2, 3, 4, 5, 6	-	-	

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.

Notes

This Certified Reference Material was originally analysed in December 1999. The certificate has now been revised to incorporate additional results, and the formatting has been changed to include derivations of uncertainty values. The batch has been made and subsequently re-certified 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 ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

This certification is applicable to the whole of the disc, although in accordance with normal practice for emission spectrometry, it is appropriate to avoid use of the middle portion of ~12mm diameter.

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 initial analysis. This certification will therefore expire in December 2019, although we reserve the right to make changes as issue revisions, in the intervening period.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.