

# CERTIFICATE OF ANALYSIS

**55X G04H2 (batch D)**

## Certified Reference Material Information

Type: ALUMINIUM/SILICON/COPPER (CAST)  
Form and Size: Disc 45mm diameter x 15mm thick  
Produced by: Nissan Motors, UK  
Certified and Supplied by: MBH Analytical Limited

## Certified Analysis

### Percentage element by weight

Element	Cu	Mg	Si	Fe	Mn	Ni	Zn	Ti
Value <sup>1</sup>	3.21	0.360	5.22	0.349	0.203	0.0104	0.185	0.0553
Uncertainty <sup>2</sup>	0.04	0.007	0.05	0.007	0.003	0.0007	0.004	0.0019

Element	Pb	Sn	Cr	P	Na	Ca	Sr
Value <sup>1</sup>	0.0159	0.0049	0.0108	(0.0020)	(0.0009)	0.0010	0.0025
Uncertainty <sup>2</sup>	0.0008	0.0009	0.0011	-	-	0.0003	0.0004

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

## Definitions

- <sup>1</sup> 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.
- <sup>2</sup> 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 31st March 2005

C Eveleigh

## **Method of Preparation**

This reference material was produced from commercial 'automotive' alloy, prepared in a 4-tonne gas-fired furnace. The melt was rotary-degassed using an argon purge with Ecoflux 143; it was then drossed-off and re-homogenised. The discs were prepared by sequentially casting from an intermediate ladle into water-cooled iron chill moulds. 2mm was removed from the cast face of each disc, to minimise any surface effects.

## **Sampling**

Samples for chemical analysis were taken from various positions throughout the casting process. At least 10% of all discs were selected for non-destructive homogeneity testing.

## **Homogeneity**

The discs were checked for sample and batch uniformity using an optical emission spectrometer. One disc was also checked for vertical uniformity. For each of the surfaces checked, the differences between the averaged result and the overall mean value were evaluated to ensure that the homogeneity of the material comprising the batch satisfied the definition given in ISO guide 30 - 1992.

Using the combined data for 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: Aluminium alloys are generally prepared by machining on a mill or a 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.

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	Cu	Mg	Si	Fe	Mn	Ni	Zn	Ti
1	3.170	0.341	5.135	0.332	0.192	0.0097	0.176	0.0511
2	3.19	0.346	5.156	0.332	0.197	0.0098	0.177	0.0523
3	3.19	0.348	5.19	0.343	0.199	0.0101	0.180	0.0528
4	3.19	0.354	5.21	0.346	0.200	0.0102	0.181	0.0549
5	3.196	0.355	5.23	0.349	0.201	0.0103	0.182	0.0551
6	3.198	0.360	5.23	0.350	0.201	0.0107	0.183	0.0552
7	3.205	0.362	5.234	0.350	0.203	0.0108	0.183	0.0557
8	3.21	0.362	5.28	0.353	0.205	0.0109	0.190	0.0559
9	3.228	0.362	5.298	0.354	0.206	0.0115	0.190	0.0560
10	3.229	0.363		0.357	0.206		0.190	0.0585
11	3.241	0.373		0.362	0.209		0.192	0.0607
12	3.25	0.376		0.364	0.210		0.193	
13	3.264	0.378			0.212			
<b>Mean</b>	<b>3.212</b>	<b>0.360</b>	<b>5.218</b>	<b>0.349</b>	<b>0.203</b>	<b>0.0104</b>	<b>0.185</b>	<b>0.0553</b>
<b>Std Dev</b>	0.028	0.011	0.053	0.010	0.006	0.0006	0.006	0.0027
<b>C<sub>(95%)</sub></b>	0.017	0.007	0.041	0.006	0.003	0.0004	0.004	0.0018

Sample	Pb	Sn	Cr	P	Na	Ca	Sr
1	0.0149	0.0036	0.0087	0.0009	0.0006	0.0007	0.0018
2	0.0150	0.0039	0.0096	0.0014	0.001	0.0008	0.0020
3	0.0152	0.0043	0.0098	0.0020	0.0012	0.0009	0.0023
4	0.0154	0.0044	0.0099	0.0021		0.0010	0.0024
5	0.0159	0.0047	0.0101	0.0023		0.0014	0.0026
6	0.0163	0.0054	0.0107	0.0024			0.0028
7	0.0169	0.0058	0.0115	0.0029			0.0028
8	0.0175	0.0067	0.0115				0.0029
9			0.0129				0.0030
10			0.0136				
<b>Mean</b>	<b>0.0159</b>	<b>0.0049</b>	<b>0.0108</b>	<b>0.0020</b>	<b>(0.0009)</b>	<b>0.0010</b>	<b>0.0025</b>
<b>Std Dev</b>	0.0009	0.0010	0.0015	0.0007	-	0.0003	0.0004
<b>C<sub>(95%)</sub></b>	0.0008	0.0009	0.0011	0.0006	-	0.0003	0.0003

Note: C<sub>(95%)</sub> 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  
Laboratory Testing Inc  
Central Iron & Steel Research Inst  
Institute of Iron & Steel Technology  
RWTUV Laboratory  
Coleshill Laboratories Ltd  
Mottrams Ltd  
Dubai Aluminium Co Limited  
Fu Shun Aluminium Smelter  
De Bruyn Spectroscopic Solutions Ltd  
Genitest Inc

Middlesbrough, England  
Sheffield, England  
Milperra, NSW, Australia  
Hatfield, PA, USA  
Beijing, China  
Shanghai, China  
Brno, Czech Republic  
Birmingham, England  
Congleton, England  
Jebel Ali, Dubai, UAE  
Fu Shun District, China  
Western Cape, South Africa  
Montreal, Canada

UKAS accreditation 0239  
UKAS accreditation 0012  
NATA accreditation 492  
A2LA accreditation 0117  
CNAL accreditation 0435  
CNAL accreditation 0783  
CAI accreditation 1060

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

## Analytical Methods Used

ELEMENT	RESULT No. & METHOD		
	ICP-AES	FAAS	OTHER
Copper	1, 5, 7-13	2, 6	3, 4 photometric (BCO, neocuprone)
Magnesium	1-4, 6-9, 12, 13	5, 10, 11	
Silicon	2, 7-9	-	3-6 gravimetric (perchloric acid)
Iron	1-5, 9-11	12	1 photometric (molybdenum blue)
Manganese	1, 2, 5, 6, 8-13	3	6-8 photometric (orthophenanthroline)
Nickel	1-4, 6, 7	5, 8, 9	4, 7 photometric (periodate)
Zinc	1-3, 6, 8-12	4, 5, 7	
Titanium	1-7, 11	9	8, 10 photometric (di-antipyryl methane)
Lead	1-3, 7, 8	4-6	
Tin	1-5, 8	6	7 photometric (phenylfluorone)
Chromium	2-5, 7-10	1, 6	
Phosphorus	1, 4-7	-	2, 3 photometric (molybdenum blue)
Sodium	1, 3	2	
Calcium	1, 3, 5	2, 4	
Strontium	2, 4-8	1, 3, 9	

## Notes

This Certified Reference Material has been produced and 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).

The combination of alloying elements used in a complex material of this type, coupled with the unidirectional solidification effects associated with semi-chill casting, has 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; material to the rear of the disc, to a depth of ~5mm, is not certified.

This material will remain stable 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 original analysis. This certification will therefore expire in March 2025, 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 preparation of this product was supervised by A Barton, Nissan Motors, UK. The testing, analysis and certification 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.