

# CERTIFICATE OF ANALYSIS

**22X 1051 (batch D)**

## Certified Reference Material Information

Type: NICKEL / CHROMIUM ALLOY (CAST)  
Form and Size: Disc 40mm Diameter x 15mm Thickness  
Produced by: Willan Metals Limited  
Certified and supplied by: MBH Analytical Limited

## Certified Analysis

### Percentage element by weight

Element	C	Si	S	Mn	Al	Cu	Co
Value <sup>1</sup>	0.166	0.58	0.0015	0.206	4.35	0.115	18.98
Uncertainty <sup>2</sup>	0.003	0.02	0.0002	0.006	0.04	0.002	0.09

Element	P	Fe	Cr	Mo	Ti	Mg	B
Value <sup>1</sup>	0.0040	0.575	15.95	4.50	1.078	0.0186	0.0009
Uncertainty <sup>2</sup>	0.0004	0.011	0.08	0.03	0.013	0.0016	0.0002

## 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 12<sup>th</sup> January 2006

C Eveleigh



## **Method of Preparation**

This reference material was produced from commercial-grade alloy. The discs are the product of one melt poured under low-pressure protective atmosphere into a single investment-casting mould with a feeding system designed to ensure sound discs. Metal was removed from the cast faces of the discs to minimise surface effects.

## **Sampling**

Samples for chemical analysis, and discs for homogeneity checks, were taken from several positions within the mould. Approximately 10% of all discs were incorporated into the schedule for 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 - 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, described 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: Nickel-base alloys are generally prepared by finishing, milling, turning or polishing. 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	C	Si	S	Mn	Al	Cu	Co
1	0.161	0.550	0.0011	0.199	4.24	0.110	18.83
2	0.164	0.556	0.0011	0.200	4.30	0.110	18.87
3	0.164	0.563	0.0012	0.200	4.33	0.112	18.87
4	0.165	0.574	0.0013	0.201	4.339	0.112	18.93
5	0.165	0.575	0.0013	0.204	4.345	0.116	18.93
6	0.165	0.579	0.0013	0.205	4.348	0.116	18.94
7	0.166	0.600	0.0014	0.210	4.36	0.116	19.04
8	0.167	0.603	0.0017	0.217	4.387	0.117	19.09
9	0.168	0.608	0.0018	0.220	4.390	0.118	19.14
10	0.168	0.610	0.0018		4.407	0.119	19.16
11	0.170		0.0020		4.434		
<b>Mean</b>	<b>0.166</b>	<b>0.582</b>	<b>0.0015</b>	<b>0.206</b>	<b>4.353</b>	<b>0.115</b>	<b>18.98</b>
<b>Std Dev</b>	0.002	0.022	0.0003	0.008	0.053	0.003	0.12
<b>C<sub>(95%)</sub></b>	0.002	0.016	0.0002	0.006	0.036	0.002	0.08

Sample	P	Fe	Cr	Mo	Ti	Mg	B
1	0.0034	0.550	15.84	4.426	1.05	0.0158	0.0005
2	0.0037	0.553	15.88	4.426	1.056	0.0168	0.0007
3	0.0040	0.565	15.88	4.489	1.074	0.0169	0.0010
4	0.0042	0.569	15.89	4.490	1.08	0.0176	0.0010
5	0.0042	0.580	15.93	4.502	1.081	0.0179	0.0011
6	0.0044	0.580	16.04	4.512	1.085	0.0199	0.0011
7		0.580	16.04	4.52	1.085	0.0200	0.0011
8		0.584	16.07	4.53	1.092	0.0211	
9		0.586		4.532	1.112	0.0214	
10		0.601		4.54			
11				4.543			
<b>Mean</b>	<b>0.0040</b>	<b>0.575</b>	<b>15.95</b>	<b>4.501</b>	<b>1.078</b>	<b>0.0186</b>	<b>0.0009</b>
<b>Std Dev</b>	0.0004	0.016	0.09	0.041	0.016	0.0020	0.0002
<b>C<sub>(95%)</sub></b>	0.0004	0.011	0.08	0.028	0.013	0.0016	0.0002

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

ATI Allvac Ltd	Sheffield, England	UKAS accreditation 0638
Bodycote Materials Testing Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL Accreditation 0367
Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Genitest Inc	Montreal, Canada	

Note: to achieve National Accreditation (eg UKAS, NATA, CNAL, NABL), 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
Carbon	-	-	2-11 1 combustion (infra-red detection) combustion (volumetric detection)
Silicon	3, 4, 5, 10	-	2, 8 1, 6, 7, 9 photometric (molybdenum blue) gravimetric (perchloric acid)
Sulfur	4	-	2, 3, 5-11 1 combustion (infra-red detection) combustion (volumetric detection)
Manganese	3, 4, 5, 7	1, 2	6, 8, 9 1 photometric (periodate)
Aluminium	1, 4, 5, 10, 11	2, 3, 6	8 7, 9 volumetric (EDTA) photometric (chrome azurol S)
Copper	2, 3, 4, 8, 10	1, 5, 6, 7, 9	1, 2, 7 4 volumetric (iodine) gravimetric (nitroso beta naphthol)
Cobalt	3, 5, 8, 9, 10	6	2 3 photometric (molybdenum blue) volumetric (alkalimetric)
Phosphorus	1, 4, 5, 6	-	3 3 volumetric (alkalimetric) photometric (sulfsalicylic acid)
Iron	1, 2, 6, 7, 8	4, 9, 10	5 2-4, 6-8 5 volumetric (dichromate) volumetric (ferrous ammonium sulfate)
Chromium	1, 5	-	1, 3 1, 3 volumetric (metavanadate)
Molybdenum	2, 5, 9, 10	4, 7	6, 8, 11 1, 3 photometric (thiocyanate)
Titanium	3, 6, 7, 9	1	2, 4 5, 8 photometric (di antipyril methane) photometric (hydrogen peroxide)
Magnesium	1, 3, 4, 6, 9	2, 5, 7, 8	
Boron	1, 3, 4, 5, 6, 7	-	2 2 photometric (circumin)

## 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 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

This certification is applicable to the whole of the disc.

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 this certificate. This certification will therefore expire in January 2026, 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.