

215X HB5 L Page 1 of 4 August 2009

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# CERTIFICATE OF ANALYSIS

# 215X HB5 (batch L)

# **Certified Reference Material Information**

Type: NICKEL HASTELLOY B-TYPE (CHILL CAST)

Form and Size: Disc 40mm Diameter x ~15mm Thickness

Manufactured by: Polycast Ltd

Certified and Supplied by: MBH Analytical Ltd

# **Assigned Values**

#### Percentage element by weight

Element	С	Si	S	Р	Mn	Cu	Fe	Cr
Value 1	0.151	1.05	0.0113	0.038	0.335	0.268	3.45	0.123
Uncertainty <sup>2</sup>	0.010	0.05	0.0007	0.002	0.006	0.007	0.10	0.003

Element	Мо	Со	V	Al	Ti	В	N	Ni
Value <sup>1</sup>	26.1	2.49	0.136	0.49	0.192	0.003	0.006	65.3
Uncertainty <sup>2</sup>	0.2	0.02	0.007	0.02	0.008	0.001	0.001	0.3

## **Definitions**

- 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.
- 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 14 <sup>th</sup> August 2009





# **Method of Preparation**

This reference material was produced from commercial-purity metals, and master alloys. The discs are the product of one melt poured into a sequence of multiple chill moulds with feeding systems designed to ensure sound discs. Approximately 2mm has been removed from the cast faces of the discs to minimise surface effects.

### **Sampling**

Samples for chemical analysis were taken from various positions throughout the casting process. At least 15% 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.

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, 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**

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:

Nickel-base alloys are generally prepared by linishing, 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**

0.18

C (95%)

0.02

			<u>Percenta</u>	ge element l	oy weight			
Sample	С	Si	S	Р	Mn	Cu	Fe	Cr
1	0.144	0.962	0.0095	0.0348	0.322	0.254	3.240	0.118
2	0.146	0.968	0.0095	0.0350	0.327	0.258	3.24	0.119
3	0.148	0.973	0.0103	0.0359	0.328	0.260	3.30	0.119
4	0.150	1.015	0.0105	0.0365	0.328	0.261	3.459	0.119
5	0.151	1.058	0.0114	0.0382	0.337	0.262	3.488	0.120
6	0.151	1.061	0.0119	0.0384	0.340	0.264	3.540	0.120
7	0.152	1.089	0.0120	0.0387	0.340	0.274	3.554	0.121
8	0.152	1.105	0.0120	0.0399	0.341	0.278	3.567	0.121
9	0.155	1.12	0.012	0.0400	0.344	0.278	3.569	0.127
10	0.155	1.137	0.0120	0.0418	0.344	0.280	3.600	0.128
11	0.156		0.0127			0.280		0.129
12	0.157							0.134
Mean	0.151	1.05	0.0113	0.0379	0.335	0.268	3.45	0.123
Std Dev	0.004	0.07	0.0011	0.0023	0.008	0.010	0.14	0.005
C <sub>(95%)</sub>	0.003	0.05	0.0007	0.0017	0.006	0.007	0.10	0.003
Sample	Мо	Со	V	AI	Ti	В	N	Ni
1	25.80	2.43	0.125	0.450	0.180	0.0022	0.0051	64.92
2	25.92	2.467	0.126	0.458	0.181	0.0023	0.0060	65.10
3	25.92	2.47	0.129	0.472	0.183	0.0024	0.0060	65.20
4	25.99	2.472	0.130	0.490	0.184	0.0027	0.0061	65.28
5	26.01	2.482	0.131	0.492	0.185	0.0034	0.0061	65.31
6	26.18	2.485	0.135	0.500	0.193	0.0039	0.0073	65.36
7	26.22	2.492	0.140	0.502	0.193	0.0040		65.40
8	26.33	2.501	0.148	0.503	0.194	0.0041		65.40
9	26.48	2.511	0.148	0.508	0.195	0.0042		65.57
10	26.50	2.520	0.148	0.508	0.201			
11		2.557			0.203			
12					0.206			
Mean	26.14	2.49	0.136	0.488	0.192	0.0032	0.0061	65.28
Std Dev	0.25	0.03	0.009	0.021	0.009	0.0008	0.0007	0.19
_	0.40	0.00	0.007	0.045	0.000	0.0000		0.45

Note:  $C_{(95\%)}$  is the 95% half-width confidence interval derived from the equation:  $C_{(95\%)} = (t \ x \ SD)/\sqrt{n}$ 

0.007

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.

0.015

0.006

0.0006

0.0007

0.15

#### **Participating Laboratories**

ATI AllVac Ltd
Inco Test Ltd
Metals Technology Testing Ltd
Sheffield Assay Office
Bodycote Materials Testing
Universal Scientific Laboratory Ltd
Genitest Inc
Institute of Iron and Steel Technology
Luo Yang Copper Co Ltd
Sargam Metals Pvt Ltd
TCR Engineering Services Pvt Ltd
Shriram Institute for Industrial Research
De Bruyn Spectroscopic Solutions Ltd
Coleshill Laboratories Ltd

Sheffield, England
Hereford, England
Sheffield, England
Sheffield, England
Middlesbrough, England
Milperra, NSW, Australia
Montreal, Canada
Shanghai, China
Luo Yang, He Nan, China
Chennai, India
Mumbai, India
Delhi, India
Johannesburg, South Africa
Birmingham, England

UKAS accreditation 1385 UKAS accreditation 0281 UKAS accreditation 0963 UKAS accreditation 0012 UKAS accreditation 0239 NATA accreditation 0492 PRI accreditation 123077 CNAL accreditation 0783 CNAL accreditation 0173 NABL accreditation 0025 NABL accreditation 0367 NABL accreditation 0045

Note: to achieve the above accreditation (eg UKAS, NATA, etc), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

#### Analytical Methods Used

ELEMENT		RES	<b>JULT No. &amp; MET</b>	HOD
	ICP-AES	FAAS		OTHER
Carbon	-	-	all	combustion (IR or volumetric detection)
Silicon	2, 10	-	1, 3-7, 9	gravimetric (perchloric acid)
			8	photometric (molybdenum blue)
Sulfur	3	-	1, 2, 4-11	combustion (IR or volumetric detection)
Phosphorus	2, 3, 6, 7, 9, 10	-	1, 5	volumetric (alkalimetric)
			4, 8	photometric (molybdenum blue)
Manganese	2-4, 7, 9	1, 8, 10	5	volumetric (arsenite)
			6	photometric (periodate)
Copper	1, 2, 7, 9, 11	3-5, 10	6	photometric (BCO)
			8	volumetric (thiosulfate)
Iron	1, 2, 7-9	3, 6, 10	4, 5	volumetric (dichromate)
Chromium	2-4, 6, 10, 12	5, 8, 9, 11	1, 7	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 3, 4, 8, 10	6, 7	2	gravimetric (α-benzoin oxime)
			5, 9	photometric (thiocyanate)
Cobalt	1, 2, 4, 6-9, 11	3, 5, 10		
Vanadium	1-5, 7, 8	6, 9, 10		
Aluminium	2, 3, 7-9	1, 5, 6	4, 10	photometric (chrome azurol S)
Titanium	2, 3, 5, 7-9, 11	1, 4, 12	6	ICP-MS
			10	photometric (di-antipyryl methane)
Boron	1-6, 8, 9	-	7	ICP-MS
Nitrogen	-	-	1-5	inert gas fusion (thermal conductivity)
			6	photometric (Nessler's reagent)
Nickel	1, 2, 8	-	3, 7, 9	volumetric (dimethyl glyoxime/EDTA)
			4, 5, 6	gravimetric (dimethyl glyoxime)

#### **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 35-2006, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The unidirectional solidification effects associated with chill 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 12mm. Material to the rear of the disc, to a depth of ~3mm, 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 this certificate. This certification will therefore expire in August 2029, although we reserve the right to make changes as issue revisions, in the intervening period.

The manufacture, analysis and certification of this product were supervised by C Eveleigh, PhD, Technical Director, MBH Analytical Ltd. This sample is also available in the form of chippings.

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