

# CERTIFICATE OF ANALYSIS

**28X 6254 (batch M)**

## Certified Reference Material Information

Type: NICKEL INCONEL 625-TYPE (CHILL CAST)  
Form and Size: Disc 40mm Diameter x 17mm Thickness  
Manufactured by: Polycast Limited  
Certified and Supplied by: MBH Analytical Limited

## Assigned Values

### Percentage element by weight

Element	C	Si	S	P	Mn	Cu	Fe	Nb
Value <sup>1</sup>	0.047	(0.79)	0.0160	0.0097	0.457	0.044	3.33	3.60
Uncertainty <sup>2</sup>	0.002	-	0.0013	0.0010	0.010	0.004	0.06	0.04

Element	Cr	Mo	Co	Ti	Al	B	Ni	N
Value <sup>1</sup>	22.71	8.92	0.195	(0.15)	(0.015)	0.005	59.55	0.0364
Uncertainty <sup>2</sup>	0.14	0.08	0.007	-	-	0.001	0.14	0.0008

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 29th October 2007

C Eveleigh



## **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. Metal was 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. One disc was checked for vertical uniformity using the same method.

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	P	Mn	Cu	Fe	Nb
1	0.0443	0.748	0.0130	0.0084	0.434	0.0367	3.245	3.53
2	0.0444	0.759	0.0138	0.0090	0.440	0.0369	3.25	3.54
3	0.045	0.780	0.014	0.0090	0.446	0.038	3.278	3.55
4	0.0459	0.782	0.015	0.0095	0.449	0.040	3.278	3.559
5	0.046	0.832	0.015	0.0098	0.454	0.0402	3.339	3.575
6	0.0462	0.845	0.0151	0.010	0.455	0.0412	3.359	3.60
7	0.0471		0.0157	0.0105	0.461	0.0478	3.39	3.617
8	0.0471		0.0171	0.0116	0.466	0.0482	3.420	3.619
9	0.0478		0.0171		0.467	0.050	3.44	3.625
10	0.0480		0.0179		0.469	0.0517		3.645
11	0.0490		0.0191		0.488	0.0540		3.67
12	0.0504		0.0196					3.704
<b>Mean</b>	<b>0.0468</b>	<b>(0.79)</b>	<b>0.0160</b>	<b>0.0097</b>	<b>0.457</b>	<b>0.044</b>	<b>3.333</b>	<b>3.603</b>
<b>Std Dev</b>	0.0018	-	0.0021	0.0010	0.015	0.006	0.074	0.054
<b>C (95%)</b>	0.0012	-	0.0013	0.0008	0.010	0.004	0.057	0.034

Sample	Cr	Mo	Co	Ti	Al	B	Ni	N
1	22.49	8.83	0.179	0.120	0.0120	0.0035	59.22	0.035
2	22.52	8.871	0.181	0.122	0.0125	0.0040	59.29	0.0352
3	22.53	8.878	0.183	0.140	0.0193	0.0041	59.51	0.0354
4	22.62	8.895	0.187	0.143		0.0053	59.58	0.0362
5	22.70	8.907	0.194	0.170		0.0056	59.60	0.0362
6	22.71	8.92	0.198	0.171		0.0061	59.62	0.037
7	22.74	9.009	0.199	0.173		0.0070	59.65	0.0371
8	22.76	9.048	0.200	0.176			59.70	0.0373
9	22.78		0.205				59.75	0.0379
10	22.92		0.208					
11	23.00		0.209					
<b>Mean</b>	<b>22.71</b>	<b>8.920</b>	<b>0.195</b>	<b>(0.15)</b>	<b>(0.015)</b>	<b>0.0051</b>	<b>59.55</b>	<b>0.0364</b>
<b>Std Dev</b>	0.16	0.073	0.011	-	-	0.0013	0.18	0.0010
<b>C (95%)</b>	0.11	0.061	0.007	-	-	0.0012	0.14	0.0008

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

ATI AllVac Ltd	Sheffield, England	UKAS accreditation 1385
Inco Test Ltd	Hereford, England	UKAS accreditation 0281
Metals Technology Testing Ltd	Sheffield, England	UKAS accreditation 0963
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
London & Scandinavian Met Co	Rotherham, England	UKAS accreditation 1091
Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
Universal Scientific Laboratory Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co Ltd	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Pvt Ltd	Mumbai, India	NABL accreditation 0367
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Genitest Inc	Montreal, Canada	

Note: to achieve National Accreditation (eg UKAS, A2LA, 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	XRF	FAAS	OTHER
Carbon	-	-	-	all combustion (infra-red detection)
Silicon	2, 6	-	-	3, 4 photometric (molybdenum blue)
				1, 5 gravimetric (perchloric acid)
Sulfur	-	-	-	all combustion (infra-red detection)
Phosphorus	1-4, 6, 8	-	-	5 photometric (molybdenum blue)
				7 volumetric (alkalimetric)
Manganese	1, 3, 5-7, 10	2	4	9, 11 photometric (periodate)
				8 volumetric (arsenite)
Copper	1-3, 6, 8, 10	9	4, 5, 7	11 photometric (BCO)
Iron	1, 3-9	-	2	
Niobium	3, 5, 7-12	2	6	4 gravimetric
				1 photometric (4-par)
Chromium	1, 6, 7, 9, 11	-	-	2-5, 8, 10 volumetric (FAS)
Molybdenum	1, 3, 7, 8	-	6	2 gravimetric ( $\alpha$ -benzoin oxime)
				4, 5 photometric (thiocyanate)
Cobalt	1, 3, 4, 7-11	-	2, 5, 6	
Titanium	2, 4, 7, 8	3	1	5, 6 photometric (DAP)
Aluminium	2	-	1, 3	
Boron	1-7	-	-	
Nickel	7, 8	-	-	1, 4 volumetric (EDTA/DMGO)
				2, 3, 5, 6, 9 gravimetric (dimethyl glyoxime)
Nitrogen	-	-	-	1-3, 5-9 inert gas fusion (thermal conductivity)
				4 photometric (Nessler's reagent)

## 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 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 ~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 this certificate. This certification will therefore expire in October 2027, 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.

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