

# CERTIFICATE OF ANALYSIS

**212X 4005 (batch G)**

## Certified Reference Material Information

Type: NICKEL/COPPER MONEL-TYPE (CHILL CAST)

Form and Size: Disc ~40mm diameter

Manufactured by: Polycast Ltd

Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	C	Si	S	P	Mn	Cu	Fe	Cr
Value <sup>1</sup>	0.0494	2.52	0.0038	0.0106	1.437	21.53	1.053	0.20
Uncertainty <sup>2</sup>	0.0017	0.02	0.0005	0.0009	0.014	0.10	0.012	0.01

Element	Mo	Nb	Co	Ti	Al	Mg	Pb	Ni
Value <sup>1</sup>	0.102	0.341	0.156	1.005	1.399	(0.0026)	0.0091	(70.1)
Uncertainty <sup>2</sup>	0.005	0.011	0.003	0.016	0.010	-	0.0012	-

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 \_\_\_\_\_

C Eveleigh

on 28<sup>th</sup> August 2017

## **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**

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. The testing procedure was in accordance with ASTM E826 and the material found acceptable.

From this test data, through-batch variation values were derived for each element as an indicator of any minor compositional variation (as determined for the specific sample size and other limitations of 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, 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**

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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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 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.

For OES, 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	Cr
1	0.0467	2.468	0.0026	0.0084	1.411	21.38	1.027	0.1848
2	0.0469	2.483	0.0030	0.0096	1.418	21.41	1.036	0.1900
3	0.0472	2.498	0.0030	0.0098	1.422	21.43	1.044	0.1946
4	0.0476	2.504	0.0030	0.0099	1.428	21.54	1.045	0.1980
5	0.0480	2.512	0.0031	0.0101	1.432	21.55	1.049	0.1990
6	0.0487	2.518	0.0033	0.0103	1.445	21.56	1.049	0.2003
7	0.0491	2.523	0.0039	0.0103	1.447	21.56	1.054	0.2017
8	0.0497	2.545	0.0041	0.0107	1.451	21.60	1.055	0.2100
9	0.0510	2.550	0.0043	0.0118	1.454	21.61	1.060	0.2110
10	0.0516	2.557	0.0044	0.0125	1.466	21.67	1.081	0.2220
11	0.0530	2.570	0.0045	0.0129			1.083	
12	0.0531	2.570	0.0047					
13			0.0053					
<b>Mean</b>	<b>0.0494</b>	<b>2.524</b>	<b>0.0038</b>	<b>0.0106</b>	<b>1.437</b>	<b>21.53</b>	<b>1.053</b>	<b>0.2011</b>
<b>Std Dev</b>	0.0023	0.0337	0.0008	0.0013	0.018	0.09	0.017	0.0109
<b>C<sub>(95%)</sub></b>	0.0015	0.0214	0.0005	0.0009	0.013	0.07	0.011	0.0078

Sample	Mo	Nb	Co	Ti	Al	Mg	Pb	Ni
1	0.0943	0.3225	0.1507	0.985	1.376	0.0006	0.0074	70.02
2	0.0963	0.3230	0.1517	0.989	1.377	0.0007	0.0075	70.02
3	0.0963	0.3250	0.1523	0.997	1.394	0.0010	0.0076	70.03
4	0.0987	0.3389	0.1544	1.007	1.394	0.0018	0.0078	70.06
5	0.1000	0.3459	0.1546	1.007	1.398	0.0019	0.0085	70.18
6	0.1003	0.3480	0.1550	1.024	1.399	0.0024	0.0103	70.20
7	0.1007	0.3493	0.1551	1.025	1.402	0.0025	0.0107	
8	0.1050	0.3571	0.1602		1.402	0.0028	0.0108	
9	0.1074	0.3600	0.1615		1.412	0.0047	0.0112	
10	0.1080		0.1620		1.414	0.0049		
11	0.1099		0.1630		1.418	0.0050		
<b>Mean</b>	<b>0.1015</b>	<b>0.3411</b>	<b>0.1564</b>	<b>1.005</b>	<b>1.399</b>	<b>(0.0026)</b>	<b>0.0091</b>	<b>70.09</b>
<b>Std Dev</b>	0.0053	0.0145	0.0044	0.016	0.014	-	0.0016	0.08
<b>C<sub>(95%)</sub></b>	0.0035	0.0112	0.0030	0.015	0.009	-	0.0012	0.09

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

Exova Ltd  
Sheffield Analytical Services  
Metals Technology (Testing) Ltd  
Genitest, Inc  
Shanghai Jinyi Test Technology Co  
Shandong Metallurgical & Science Research  
TCR Engineering Services Ltd  
Raghavendra Spectrometallurgical Lab.  
Instytut Metalurgii Zelaza  
Tec-Eurolab  
Degerfors Laboratorium AB  
INCDMNR-IMNR  
Coleshill Laboratories Ltd  
Mineral & Metallurgical Laboratories  
AMG Superalloys UK Ltd  
Analyticka Laborator Lithea sro

Middlesbrough, England  
Sheffield, England  
Sheffield, England  
Montreal, Canada  
Shanghai, China  
Jinan, Shandong, China  
Mumbai, India  
Bangalore, India  
Gliwice, Poland  
Campogalliano, Italy  
Degerfors, Sweden  
Pantelimon, Romania  
Birmingham, England  
Bangalore, India  
Rotherham, England  
Brno, Czech Republic

UKAS accreditation 0239  
UKAS accreditation 0012  
UKAS accreditation 0963  
PJ accreditation 95510  
CNAL accreditation 0783  
CNAS accreditation 1461  
NABL accreditation 0367  
NABL accreditation 0371  
PCA accreditation AB554  
ACCREDIA accreditation 52  
SWEDAC accreditation 1890

Note: to achieve the above accreditation (eg UKAS, etc), 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
Carbon	-	-	all combustion (infra-red detection)
Silicon	1, 4, 5, 7, 10, 11	-	2, 9 photometric (molybdenum blue)
Sulfur	1, 6, 9	-	3, 6, 8, 12 gravimetric (perchloric acid)
Phosphorus	1-5, 7, 8, 10	-	2-5, 7, 8, 10-13 combustion (infra-red detection)
Manganese	2-8	-	6 volumetric (alkalimetric)
Copper	2, 4, 5, 8-10	-	9, 11 photometric (molybdenum blue)
Iron	1-5, 8, 9, 11	-	1 photometric (periodate)
Chromium	1-4, 6, 7	8	9, 10 volumetric (arsenite)
Molybdenum	1, 3, 4, 6, 7, 9, 10	11	1 photometric (BCO)
Niobium	1, 2, 4, 5, 7, 8	-	3, 6 volumetric (thiosulfate)
Cobalt	2-9	1	7 electrogravimetric
Titanium	1, 3-5, 7	-	6, 10 volumetric (dichromate)
Aluminium	1, 2, 4, 6-9	3	7 photometric (sulfsalicylic acid)
Magnesium	1-10	11	5, 9 volumetric (ferrous ammonium sulfate)
Lead	1-5, 7-9	-	10 photometric (diphenyl carbazide)
Nickel	4, 5	-	2 gravimetric (8-hydroxy quinoline)
			5, 8 photometric (thiocyanate)
			3, 9 photometric (chlorosulfophenol)
			6 gravimetric
			10 photometric (2β naphthol)
			11 gravimetric
			2, 6 photometric (diantipyryl methane)
			5, 11 photometric (chrome azurol S)
			10 gravimetric
			6 gravimetric (sulfate)
			1, 2 gravimetric (dimethyl glyoxime)
			3, 6 photometric (dimethyl glyoxime)

## Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The unidirectional solidification effects associated with this method of 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 indefinitely, 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. Technical support for this certification will therefore expire in August 2037, although we reserve the right to make changes as issue revisions, in the intervening period.

The production, 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.