

CERTIFICATE OF ANALYSIS

28X 6252 (batch Q)

Certified Reference Material Information

Type: NICKEL INCONEL 625-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	Cr	Mo	Co
Value ¹	0.078	0.982	0.0144	0.0110	0.331	20.33	8.78	0.279
Uncertainty ²	0.003	0.018	0.0011	0.0007	0.006	0.13	0.06	0.004

Element	Cu	Fe	Al	Ti	Nb	Ta	Ni	N
Value ¹	0.230	4.73	0.130	0.113	3.14	(0.004)	60.62	0.085
Uncertainty ²	0.004	0.05	0.002	0.003	0.04	-	0.13	0.003

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

Definitions

- ¹ The above 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.
- ² 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 14th July 2018

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	Cr	Mo	Co
1	0.0721	0.9400	0.0116	0.0093	0.3160	20.133	8.637	0.2702
2	0.0739	0.9571	0.0122	0.0104	0.3168	20.158	8.667	0.2718
3	0.0755	0.9610	0.0128	0.0105	0.3180	20.190	8.678	0.2722
4	0.0757	0.9660	0.0131	0.0106	0.3223	20.248	8.748	0.2727
5	0.0766	0.9726	0.0139	0.0108	0.3305	20.310	8.777	0.2760
6	0.0787	0.9763	0.0144	0.0108	0.3310	20.314	8.786	0.2768
7	0.0793	0.9765	0.0146	0.0109	0.3330	20.320	8.822	0.2815
8	0.0797	0.9766	0.0147	0.0111	0.3366	20.320	8.833	0.2834
9	0.0801	1.0100	0.0155	0.0113	0.3384	20.448	8.834	0.2840
10	0.0804	1.0105	0.0155	0.0121	0.3400	20.490	8.844	0.2850
11	0.0810	1.0120	0.0157	0.0131	0.3402	20.495	8.853	0.2852
12		1.0190	0.0158		0.3441	20.499	8.874	0.2860
13			0.0158					0.2868
14			0.0159					
Mean	0.0775	0.9815	0.0144	0.0110	0.3306	20.327	8.780	0.2794
Std Dev	0.0030	0.0254	0.0015	0.0010	0.0100	0.132	0.080	0.0062
C (95%)	0.0020	0.0162	0.0008	0.0007	0.0063	0.084	0.051	0.0037

Sample	Cu	Fe	Al	Ti	Nb	Ta	Ni	N
1	0.2204	4.669	0.1243	0.1075	3.096	0.0021	60.50	0.0790
2	0.2206	4.670	0.1279	0.1101	3.100	0.0024	60.50	0.0803
3	0.2209	4.673	0.1280	0.1103	3.118	0.0035	60.59	0.0804
4	0.2242	4.688	0.1285	0.1103	3.127	0.0035	60.62	0.0842
5	0.2290	4.716	0.1310	0.1108	3.130	0.0044	60.66	0.0846
6	0.2290	4.721	0.1311	0.1114	3.133	0.0060	60.68	0.0851
7	0.2310	4.725	0.1313	0.1130	3.143	0.0063	60.71	0.0857
8	0.2330	4.725	0.1314	0.1132	3.144		60.73	0.0887
9	0.2346	4.734	0.1324	0.1160	3.153			0.0893
10	0.2369	4.738	0.1350	0.1170	3.154			0.0901
11	0.2371	4.762		0.1180	3.198			
12	0.2377	4.765			3.222			
13	0.2386	4.785						
14		4.803						
Mean	0.2302	4.727	0.1301	0.1125	3.143	0.0040	60.62	0.0847
Std Dev	0.0068	0.042	0.0030	0.0033	0.037	0.0016	0.09	0.0039
C (95%)	0.0041	0.024	0.0021	0.0022	0.023	0.0015	0.07	0.0030

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	Middlesbrough, England	UKAS accreditation 0239
Sheffield Analytical Services	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Genitest, Inc	Montreal, Canada	PJ accreditation L17-153
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
INCDMNR-IMNR	Pantelimon, Romania	
PT Geoservices Ltd	Cikarang, Indonesia	
Mineral & Metallurgical Laboratories	Bangalore, India	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (UKAS, etc), 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	-	-	all combustion (infra-red detection)
Silicon	1, 2, 5, 8-12	-	3 photometric (molybdenum blue)
Sulfur	4, 13	-	4, 6, 7 gravimetric (perchloric acid)
Phosphorus	1, 2, 4-8, 10, 11	-	1-3, 5-12 combustion (infra-red detection)
Manganese	1, 2, 4-6, 8-10, 12	11	3 volumetric (alkalimetric)
Chromium	1, 3-6, 10	7	9 photometric (molybdenum blue)
Molybdenum	2-6, 9-12	-	3, 7 volumetric (arsenite)
Cobalt	1-9, 11	13	2, 8, 9, 11, 12 volumetric (ferrous ammonium sulfate)
Copper	3-5, 9-13	1, 2	1, 8 photometric (thiocyanate)
Iron	1-3, 5, 8-10, 12, 14	6, 13	7 gravimetric (α benzoin oxime)
Aluminium	1, 2, 4, 6-10	-	10 photometric (5-Cl-PADAB)
Titanium	1, 3-6, 8-10	2	12 gravimetric
Niobium	1, 2, 4-9, 11	-	6, 7 photometric (BCO)
Tantalum	1-7	-	8 electrogravimetric
Nickel	2, 3, 5	-	4 volumetric (dichromate)
Nitrogen	-	-	7, 11 photometric (sulfosalicylic acid)
			3 volumetric (EDTA)
			5 photometric (chrome azurol)
			7, 11 photometric (DAP)
			3, 10 photometric (chlorosulfophenol)
			12 gravimetric (cupferron)
			1, 6, 7 gravimetric (dimethyl glyoxime)
			4, 8 photometric (dimethyl glyoxime)
			1, 3-8 inert gas fusion (thermal conductivity)
			2, 9, 10 photometric (Nessler reagent)

Notes

This Certified Reference Material has been produced and certified 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 July 2038, 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.