

CERTIFICATE OF ANALYSIS

28X 6253 (batch T)

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	Cu
Value ¹	0.116	1.32	0.0106	0.0242	0.490	21.91	7.82	0.39	0.306
Uncertainty ²	0.002	0.03	0.0010	0.0015	0.009	0.08	0.04	0.01	0.006

Element	Fe	V	W	Al	Ti	Nb	Ta	Ni	N
Value ¹	5.62	0.052	0.0134	(0.19)	0.247	4.50	0.039	(56.8)	0.092
Uncertainty ²	0.03	0.002	0.0012	-	0.006	0.04	0.002	-	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 _____

on 9th August 2017

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. 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	Cu
1	0.1108	1.252	0.0090	0.0223	0.4705	21.70	7.748	0.3633	0.2913
2	0.1120	1.264	0.0090	0.0223	0.4720	21.84	7.785	0.3675	0.3010
3	0.1150	1.272	0.0090	0.0225	0.4779	21.85	7.786	0.3680	0.3030
4	0.1160	1.274	0.0092	0.0228	0.4795	21.88	7.806	0.3713	0.3054
5	0.1160	1.286	0.0094	0.0232	0.4820	21.89	7.812	0.3900	0.3070
6	0.1180	1.299	0.0094	0.0234	0.4876	21.90	7.816	0.3903	0.3079
7	0.1180	1.306	0.0095	0.0251	0.4892	21.90	7.822	0.3977	0.3081
8	0.1183	1.331	0.0103	0.0253	0.4954	21.94	7.822	0.3977	0.3134
9	0.1190	1.354	0.0117	0.0275	0.5050	22.07	7.826	0.4002	0.3139
10	0.1191	1.373	0.0119	0.0279	0.5050	22.08	7.849	0.4005	
11	0.1192	1.377	0.0119		0.5051		7.866	0.4030	
12		1.396	0.0120		0.5080		7.899	0.4170	
13			0.0128					0.4203	
14			0.0137						
Mean	0.1164	1.315	0.0106	0.0242	0.4898	21.91	7.820	0.3913	0.3057
Std Dev	0.0029	0.049	0.0016	0.0021	0.0137	0.11	0.039	0.0186	0.0069
C (95%)	0.0019	0.031	0.0009	0.0015	0.0087	0.08	0.025	0.0113	0.0053

Sample	Fe	V	W	Al	Ti	Nb	Ta	Ni	N
1	5.562	0.0474	0.0111	0.1802	0.2380	4.445	0.0347	56.56	0.0885
2	5.574	0.0478	0.0120	0.1830	0.2415	4.449	0.0361	56.64	0.0892
3	5.586	0.0487	0.0125	0.1861	0.2422	4.454	0.0373	56.72	0.0900
4	5.586	0.0488	0.0127	0.1871	0.2427	4.455	0.0378	56.75	0.0916
5	5.595	0.0498	0.0133	0.1931	0.2450	4.496	0.0388	57.09	0.0921
6	5.612	0.0522	0.0141	0.1956	0.2455	4.501	0.0398	57.10	0.0950
7	5.621	0.0527	0.0148	0.1989	0.2478	4.507	0.0399		0.0950
8	5.623	0.0530	0.0148	0.1990	0.2500	4.514	0.0403		0.0982
9	5.629	0.0534	0.0149	0.2020	0.2505	4.520	0.0408		
10	5.639	0.0565		0.2023	0.2508	4.572	0.0414		
11	5.659	0.0568		0.2027	0.2510	4.587			
12	5.683			0.2077	0.2530				
13	5.700				0.2560				
Mean	5.621	0.0516	0.0134	0.1948	0.2472	4.500	0.0387	56.81	0.0924
Std Dev	0.042	0.0033	0.0014	0.0089	0.0052	0.048	0.0022	0.23	0.0034
C (95%)	0.025	0.0022	0.0011	0.0056	0.0032	0.032	0.0016	0.24	0.0028

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
Genitest, Inc	Montreal, Canada	PJLA accreditation 95510
Shanghai Jinyi Test Technology Co	Shanghai, China	CNAL accreditation 0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation 0371
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
TEC-Eurolab SRL	Campogalliano, Italy	ACCREDIA accreditation 52
Degerfors Laboratorium AB	Degerfors, Sweden	SWEDAC accreditation 1890
INCDMNR-IMNR	Pantelimon, Romania	
Mineral & Metallurgical Laboratories	Bangalore, India	
AMG superalloys UK Ltd	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (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	2-4, 6, 8-11	-	1, 7 gravimetric (perchloric acid)
Sulfur	6, 7	-	5, 12 photometric (molybdenum blue)
Phosphorus	2-6, 8, 10	-	1-5, 8-14 combustion (infra-red detection)
Manganese	3-8, 10, 11	1	1, 9 photometric (molybdenum blue)
Chromium	1-3, 5-8	-	7 volumetric (alkalimetric)
Molybdenum	1-4, 6, 9-11	8	2 photometric (periodate)
Cobalt	1-9, 13	-	9, 12 volumetric (arsenite)
Copper	1, 4, 6-9	-	4, 9, 10 volumetric (ferrous ammonium sulfate)
Iron	103, 5, 7-9, 11, 12	13	5 gravimetric (α benzoïn oxime)
Vanadium	1-3, 6, 8-11	4	7, 12 photometric (thiocyanate)
Tungsten	2-8	-	10, 11 photometric (5-Cl-PADAB)
Aluminium	1, 3-7, 10, 12	11	12 gravimetric
Titanium	1-7, 9, 10, 13	-	2, 5 photometric (BCO)
Niobium	1, 2, 4, 6-9, 11	-	3 volumetric (thiosulfate)
Tantalum	1-7, 9, 10	-	4, 6 volumetric (dichromate)
Nickel	4-6	-	10 photometric (sulfsalicylic acid)
Nitrogen	-	-	5, 7 volumetric (ferrous ammonium sulfate)
			1, 9 photometric (thiocyanate)
			2, 9 photometric (chrome azurol S)
			8 gravimetric
			8, 11, 12 photometric (DAP)
			3, 10 photometric (chlorosulfophenol)
			5 gravimetric (cupferron)
			8 photometric (pyrogallol)
			1 photometric (dimethyl glyoxime)
			2, 3 gravimetric (dimethyl glyoxime)
			1, 8 photometric (Nessler reagent)
			2-7 inert gas fusion (thermal conductivity)

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 10mm. Material to the rear of the disc, to a depth of ~5mm, is not certified.

The aluminium in this sample is not evenly distributed and therefore can not be 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 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.