MBH ANALYTICAL LTD

HOLLAND HOUSE • QUEENS ROAD • BARNET • EN5 4DJ • ENGLAND • TEL: +44 (0)20 8441 2024 • FAX: +44 (0)20 8449 0810

email: info@mbh.co.uk web: www.mbh.co.uk

CERTIFICATE OF ANALYSIS

219X 20500 (batch C)

Certified Reference Material Information

Type: NICKEL / CHROMIUM ALLOY (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	Co
Value [1]	**0.0212**	**1.289**	**0.014**	**0.0049**	**0.300**	**0.0105**	**0.0111**
Uncertainty [2]	0.0011	0.014	0.001	0.0004	0.007	0.0006	0.0008

Element	Nb	Fe	Cr	Mo	Ni	W	N
Value [1]	**0.0119**	**1.517**	**51.0**	**0.0103**	**45.5**	**0.0085**	**0.199**
Uncertainty [2]	0.0012	0.014	0.2	0.0010	0.2	0.0012	0.003

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:

on 17th January 2017

MBH ANALYTICAL LIMITED _____

C. Eveleigh

Method of Preparation

This reference material was produced from commercial alloy to UNS R20500, with the minor and trace elements added as 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

Milled samples for chemical analysis were taken from several positions within the batch. In addition, at least 15% of all discs were selected for homogeneity checking.

Homogeneity

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Multiple measurements were taken from each surface under test.

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 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, 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 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.

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	Co
1	0.0190	1.2598	0.0117	0.0041	0.2798	0.0095	0.0091
2	0.0195	1.2740	0.0123	0.0043	0.2863	0.0095	0.0099
3	0.0201	1.2760	0.0123	0.0044	0.2948	0.0098	0.0101
4	0.0203	1.2772	0.0123	0.0045	0.2997	0.0100	0.0101
5	0.0203	1.2830	0.0133	0.0047	0.3000	0.0100	0.0105
6	0.0209	1.2860	0.0134	0.0048	0.3010	0.0102	0.0107
7	0.0215	1.2964	0.0134	0.0050	0.3010	0.0103	0.0110
8	0.0217	1.3010	0.0144	0.0051	0.3024	0.0110	0.0119
9	0.0225	1.3044	0.0145	0.0052	0.3062	0.0113	0.0119
10	0.0227	1.3100	0.0145	0.0053	0.3110	0.0113	0.0121
11	0.0229	1.3110	0.0146	0.0053	0.3127	0.0123	0.0122
12	0.0234		0.0151	0.0056			0.0123
13			0.0152				0.0123
14			0.0152				0.0126
Mean	**0.0212**	**1.2890**	**0.0137**	**0.0049**	**0.2995**	**0.0105**	**0.0111**
Std Dev	0.0014	0.0167	0.0012	0.0005	0.0097	0.0009	0.0011
C (95%)	0.0009	0.0112	0.0007	0.0003	0.0065	0.0006	0.0007

Sample	Nb	Fe	Cr	Mo	Ni	W	N
1	0.0099	1.4911	50.625	0.0099	45.304	0.0077	0.1920
2	0.0100	1.5010	50.751	0.0100	45.350	0.0078	0.1960
3	0.0100	1.5020	50.780	0.0101	45.382	0.0078	0.1980
4	0.0103	1.5020	50.785	0.0101	45.400	0.0080	0.1990
5	0.0108	1.5110	50.985	0.0102	45.411	0.0083	0.1991
6	0.0115	1.5157	51.090	0.0103	45.467	0.0085	0.1991
7	0.0129	1.5230	51.116	0.0105	45.497	0.0087	0.1997
8	0.0131	1.5248	51.205	0.0105	45.523	0.0092	0.2005
9	0.0132	1.5300	51.210	0.0105	45.526	0.0093	0.2038
10	0.0135	1.5310	51.290	0.0106	45.540	0.0101	
11	0.0136	1.5323	51.407	0.0106	45.577		
12	0.0138	1.5380			45.630		
Mean	**0.0119**	**1.5168**	**51.022**	**0.0103**	**45.467**	**0.0085**	**0.1986**
Std Dev	0.0016	0.0152	0.255	0.0003	0.098	0.0008	0.0032
C (95%)	0.0010	0.0097	0.171	0.0002	0.063	0.0006	0.0025

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, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
Metals Technology (Testing) Ltd	Sheffield, UK	UKAS accreditation	0963
Genitest Inc.	Montreal, QC, Canada	PRI accreditation	123077
Laboratory Testing, Inc.	Hatfield, PA, USA	A2LA accreditation	0117
Shanghai JinYi Test Technology Co. Ltd	Shanghai, China	CNAL accreditation	0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation	1461
Bureau Veritas CPS Ltd	Chennai, India	NABL accreditation	0025
TCR Engineering Services PVT. Ltd	Mumbai, India	NABL accreditation	0367
Raghavendra Spectro Metallurgical Laboratory	Bangalore, India	NABL accreditation	0371
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation	AB554
TEC Eurolab SRL	Modena, Italy	ACCREDIA accreditation	52
Mineral and Metallurgical Laboratories	Bangalore, India		
AMG Superalloys UK Ltd	Rotherham, UK		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
Coleshill Laboratories Limited	Coleshill, UK		

Note: to achieve the above-noted 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	-	-	1-12	combustion (infra-red detection)
Silicon	2-5, 9	-	1, 7, 8, 10, 11	gravimetric (perchloric acid)
			6	photometric (molybdenum blue)
Sulfur	2, 10	-	1, 3-9, 11-14	combustion (infra-red detection)
Phosphorus	1, 3-6, 8, 12	-	2, 9, 11	volumetric (alkalimetric)
			7, 10	photometric (molybdenum blue)
Manganese	1-4, 8, 11	5	6, 10	volumetric (arsenite, FAS)
			7, 9	photometric (periodate)
Copper	1, 2, 5, 6, 8, 10, 11	9	3, 4	photometric (BCO)
			7	volumetric (thiosulfate)
Cobalt	1, 3-6, 8-10, 13	7, 12	2	volumetric (iodine)
			11	photometric (2beta-naphtol)
Niobium	1, 3-12	-	2	photometric (chlorosulfophenol)
Iron	1, 2, 4, 6-8, 10, 12	-	3, 11	photometric (orthophenanthroline)
			5, 9	volumetric (dichromate)
Chromium	3, 6, 10	-	1, 2, 4, 5, 7-9, 11	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 3, 4, 7-9, 11	2	5, 6, 10	photometric (thiocyanate)
Nickel	4, 7, 9-12	-	1, 2, 5, 8	gravimetric (dimethyl glyoxime)
			3, 6	volumetric (dimethyl glyoxime, EDTA)
Tungsten	1-4, 6-10	-	5	photometric (thiocyanate)
Nitrogen	-	-	1, 2, 5, 6, 8, 9	inert gas fusion (thermal conductivity)
			3, 7	volumetric (sulfuric acid)
			4	photometric (Nessler reagent)

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2015 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 this method of chill casting, have led to the formation of inhomogeneous segregates in the rear portion of the disc. The above certification is therefore only applicable from the front face of the disc. Material to the rear of the disc, to a depth of ~3 mm, 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 January 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.