MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

13X 12534 (batch V)

Certified Reference Material Information

Type: AUSTENITIC STAINLESS STEEL (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	Ni	Cr	Mo
Value [1]	**0.069**	**0.661**	**0.0146**	**0.0281**	**0.833**	**9.27**	**18.54**	**2.309**
Uncertainty [2]	0.002	0.006	0.0004	0.0011	0.008	0.04	0.03	0.013

Element	Cu	Co	V	Nb	Al	Ti	Ta	N
Value [1]	**0.070**	**0.1018**	**0.093**	**0.193**	**0.072**	**0.19**	**0.021**	**0.069**
Uncertainty [2]	0.002	0.0012	0.002	0.006	0.002	0.01	0.002	0.002

Definitions

[1] 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.

[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 3rd July 2014

MBH ANALYTICAL LIMITED _____

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

The discs were checked for sample and batch uniformity using an optical emission spectrometer.

Using the combined 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. In addition, some of the results derived as part of this testing programme have 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:	Steels are generally prepared by linishing, grinding, turning or milling. 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. The recommended sample size is at least five replicate analyses. 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	Ni	Cr	Mo
1	0.0656	0.646	0.0132	0.0248	0.817	9.171	18.46	2.269
2	0.0658	0.649	0.0135	0.0253	0.818	9.215	18.50	2.269
3	0.0665	0.653	0.0136	0.0267	0.820	9.223	18.50	2.285
4	0.0672	0.656	0.0137	0.0273	0.824	9.223	18.50	2.302
5	0.0680	0.657	0.0142	0.0276	0.826	9.230	18.51	2.302
6	0.0684	0.658	0.0145	0.0278	0.828	9.230	18.52	2.305
7	0.0685	0.659	0.0145	0.0281	0.837	9.240	18.52	2.309
8	0.0688	0.659	0.0145	0.0286	0.838	9.261	18.55	2.314
9	0.0692	0.669	0.0146	0.0286	0.838	9.290	18.55	2.316
10	0.0693	0.671	0.0150	0.0288	0.841	9.297	18.55	2.320
11	0.0699	0.672	0.0150	0.0300	0.845	9.300	18.56	2.324
12	0.0709	0.672	0.0152	0.0305	0.849	9.300	18.58	2.331
13	0.0721	0.678	0.0154	0.0313	0.850	9.335	18.59	2.333
14	0.0728		0.0154			9.373	18.59	2.350
15	0.0730		0.0155			9.382	18.60	
16			0.0156				18.60	
Mean	**0.0691**	**0.661**	**0.0146**	**0.0281**	**0.833**	**9.271**	**18.54**	**2.309**
Std Dev	0.0024	0.010	0.0008	0.0019	0.012	0.061	0.04	0.023
C (95%)	0.0013	0.006	0.0004	0.0011	0.007	0.034	0.02	0.013

Sample	Cu	Co	V	Nb	Al	Ti	Ta	N
1	0.0664	0.0993	0.0864	0.178	0.0692	0.183	0.0175	0.0660
2	0.0667	0.0997	0.0884	0.179	0.0706	0.183	0.0186	0.0665
3	0.0667	0.1001	0.0892	0.181	0.0712	0.185	0.0195	0.0668
4	0.0671	0.1010	0.0913	0.185	0.0713	0.190	0.0195	0.0668
5	0.0671	0.1010	0.0914	0.194	0.0717	0.191	0.0204	0.0688
6	0.0684	0.1016	0.0915	0.195	0.0718	0.193	0.0209	0.0699
7	0.0686	0.1020	0.0921	0.196	0.0722	0.196	0.0224	0.0708
8	0.0687	0.1023	0.0929	0.198	0.0722	0.197	0.0240	0.0709
9	0.0692	0.1033	0.0942	0.200	0.0724	0.198	0.0251	0.0712
10	0.0717	0.1035	0.0957	0.203	0.0730	0.198		0.0714
11	0.0719	0.1039	0.0960	0.204	0.0735	0.198		
12	0.0720	0.1043	0.0971	0.205	0.0736	0.200		
13	0.0721		0.0994		0.0740			
14	0.0726				0.0745			
15	0.0739							
Mean	**0.0695**	**0.1018**	**0.0927**	**0.193**	**0.0722**	**0.193**	**0.0209**	**0.0689**
Std Dev	0.0026	0.0017	0.0037	0.010	0.0014	0.006	0.0025	0.0022
C (95%)	0.0014	0.0011	0.0022	0.006	0.0008	0.004	0.0019	0.0016

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 Assay Office	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Bureau Veritas CPS Pvt	Chennai, India	NABL accreditation 0025
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Genitest Inc	Montreal, Canada	PRI accreditation 123077
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
London & Scandinavian Met Co	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Lithea sro	Brno, Czech Republic	
Mineral & Metallurgical Laboratories	Bangalore, India	

Note: to achieve the above accreditation (UKAS, NATA, 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 (IR or volumetric detection)
Silicon	3-5, 7, 9	-	1, 2, 6, 8, 12, 13	gravimetric (perchloric acid)
			10, 11	photometric (molybdenum blue)
Sulfur	1, 15	-	2-14, 16	combustion (IR or volumetric detection)
Phosphorus	2, 4-12	-	1, 3	photometric (molybdenum blue)
			13	volumetric (alkalimetric)
Manganese	1, 3, 4, 6, 8-12	5, 13	2	photometric (periodate)
			7	volumetric (arsenite, FAS)
Nickel	2, 6, 8, 10, 12-14	3	1, 4, 5, 7, 9, 11	gravimetric (dimethyl glyoxime)
			15	volumetric (dimethyl glyoxime, EDTA)
Chromium	1, 2, 5, 6, 10, 11, 15, 16	-	3, 4, 7-9, 12-14	volumetric (ferrous ammonium sulfate)
Molybdenum	2, 4-7, 9, 12-14	10, 11	1	gravimetric (α benzoin oxime)
			3, 8	photometric (thiocyanate)
Copper	1, 3-11, 14	2, 12, 15	13	photometric (BCO)
Cobalt	1, 4-7, 10-12	2, 3, 9	8	volumetric (iodine)
Vanadium	1, 2, 5, 7, 9-11, 13	3, 6, 8	4, 12	volumetric (ferrous ammonium sulfate)
Niobium	1, 2, 4-9, 11	3	10	ICP-MS
			12	photometric (chlorosulfophenol)
Aluminium	1, 2, 4-6, 8-10, 14	7, 11, 13	3, 12	photometric (chrome azurol-S)
Titanium	1-4, 6, 7, 10, 11	9, 12	5, 8	photometric (di-antipyryl methane)
Tantalum	1, 3-9	-	2	ICP-MS
Nitrogen	-	-	3, 4, 6, 7, 9	inert gas fusion (thermal conductivity)
			1, 2, 8	volumetric (hydrochloric acid)
			5	photometric (Nessler Reagent)

Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 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 semi-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 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 2034, 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.