MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

13X 31635 (batch A)

Certified Reference Material Information

Type: AUSTENITIC STAINLESS STEEL (WROUGHT)

Form and Size: Disc, ~40mm diameter

Manufactured by: Olarra, Spain

Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	Mn	P	S	Cr	Mo	Ni
Value [1]	**0.0254**	**0.487**	**1.807**	**0.0352**	**0.0301**	**16.98**	**2.089**	**10.80**
Uncertainty [2]	0.0009	0.014	0.013	0.0006	0.0007	0.08	0.017	0.06

Element	Al	Co	Cu	Nb	Ti	V	W	N
Value [1]	**0.0054**	**0.174**	**0.404**	**0.010**	**0.149**	**0.0584**	**0.031**	**0.020**
Uncertainty [2]	0.0005	0.003	0.007	0.001	0.003	0.0008	0.001	0.001

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 20th May 2016

MBH ANALYTICAL LIMITED _____

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Method of Preparation

This reference material was produced from commercial bar with a composition similar to UNS S31635. The steel was arc-furnace melted and AOD treated prior to continuous-casting and hot rolling to final dimension.

Sampling

Samples for chemical analysis were taken from various positions throughout the batch. Approximately 10% 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. 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 a national authority. It is part of the 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 primary 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.

For optical emission spectroscopy, 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	Mn	P	S	Cr	Mo	Ni
1	0.0236	0.455	1.790	0.0339	0.0276	16.80	2.045	10.72
2	0.0241	0.461	1.795	0.0344	0.0286	16.87	2.060	10.72
3	0.0241	0.462	1.799	0.0348	0.0290	16.87	2.060	10.73
4	0.0245	0.483	1.799	0.0349	0.0296	16.90	2.069	10.75
5	0.0249	0.485	1.803	0.0349	0.0300	16.96	2.072	10.75
6	0.0253	0.495	1.804	0.0351	0.0301	16.99	2.080	10.77
7	0.0255	0.499	1.809	0.0354	0.0301	17.01	2.091	10.82
8	0.0258	0.500	1.810	0.0354	0.0302	17.02	2.100	10.82
9	0.0259	0.503	1.810	0.0356	0.0303	17.03	2.108	10.83
10	0.0262	0.504	1.813	0.0359	0.0304	17.07	2.108	10.85
11	0.0264	0.509	1.817	0.0361	0.0313	17.14	2.113	10.92
12	0.0266		1.837	0.0362	0.0314	17.15	2.124	10.93
13	0.0271				0.0316		2.129	
14					0.0316			
Mean	**0.0254**	**0.487**	**1.807**	**0.0352**	**0.0301**	**16.98**	**2.089**	**10.80**
Std Dev	0.0011	0.019	0.012	0.0007	0.0012	0.11	0.027	0.07
C (95%)	0.0007	0.013	0.008	0.0004	0.0007	0.07	0.016	0.05

Sample	Al	Co	Cu	Nb	Ti	V	W	N
1	0.0044	0.169	0.397	0.0090	0.1430	0.0568	0.0300	0.0184
2	0.0050	0.172	0.400	0.0090	0.1443	0.0569	0.0303	0.0190
3	0.0051	0.174	0.400	0.0095	0.1444	0.0571	0.0304	0.0192
4	0.0051	0.175	0.402	0.0096	0.1450	0.0572	0.0306	0.0194
5	0.0052	0.175	0.403	0.0099	0.1476	0.0574	0.0306	0.0197
6	0.0057	0.176	0.403	0.0101	0.1485	0.0583	0.0311	0.0205
7	0.0058	0.176	0.405	0.0102	0.1500	0.0587	0.0313	0.0209
8	0.0061	0.180	0.405	0.0103	0.1501	0.0592	0.0316	
9	0.0064	0.180	0.405	0.0104	0.1510	0.0595	0.0335	
10			0.407	0.0105	0.1511	0.0597		
11			0.408	0.0105	0.1530	0.0597		
12			0.410		0.1530	0.0599		
13			0.412		0.1540			
14					0.1566			
Mean	**0.0054**	**0.175**	**0.404**	**0.0099**	**0.1494**	**0.0584**	**0.0310**	**0.0196**
Std Dev	0.0006	0.004	0.004	0.0006	0.0041	0.0012	0.0011	0.0009
C (95%)	0.0005	0.003	0.003	0.0004	0.0024	0.0007	0.0008	0.0008

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
Metals Technology (Testing) Ltd.	Sheffield, UK	UKAS accreditation	0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation	0492
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
AMG Superalloys UK Ltd	Rotherham, UK		
Mineral and Metallurgical Laboratories	Bangalore, India		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
PT Geoservices Ltd.	Cikarang, Indonesia		
Coleshill Laboratories Ltd	Coleshill, UK		

Note: to achieve the above-noted accreditation (eg UKAS, NATA, 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	4, 6, 9, 11	-	1, 7, 8, 10	gravimetric (perchloric acid)
			2, 3, 5	photometric (molybdenum blue)
Manganese	3, 5-7, 9, 12	4, 8	2, 10	volumetric (arsenite, fas)
			1, 11	photometric (periodate)
Phosphorus	1, 2, 4, 6, 8, 10	-	7, 11, 12	volumetric (alkalimetric)
			3, 5, 9	photometric (molybdenum blue)
Sulfur	5, 7	-	1-4, 6, 8-14	combustion (IR or volumetric detection)
Chromium	1, 2, 6, 9-11	-	3-5, 8, 12	volumetric (ferrous ammonium sulfate)
			7	photometric (diphenyl carbazide)
Molybdenum	1, 5-7, 10, 12, 13	3, 8	2, 4, 9, 11	photometric (thiocyanate)
Nickel	3, 4, 9, 11, 12	6	1, 2, 6, 7	gravimetric (dimethyl glyoxime)
			8, 10	photometric (dimethyl glyoxime)
Aluminium	4-7, 9	1, 8	2, 3	photometric (chrome azurol s)
Cobalt	2-5, 7	6, 8, 9	1	photometric (2beta-naphtol)
Copper	2, 4, 6-8, 10, 12	3, 5, 9	1, 13	photometric (BCO)
			11	volumetric (thiosulfate)
Niobium	1-6, 9-10	7	11	photometric (chlorosulfophenol)
			8	gravimetric
Titanium	1-3, 5, 8, 10, 13, 14	4, 6, 7	9, 11, 12	photometric (DAP, peroxide)
Vanadium	1, 2, 6-8, 10, 12	4, 5, 9	3, 11	volumetric (ferrous ammonium sulfate)
Tungsten	1-4, 8, 9	6	5	photometric (thiocyanate)
			7	gravimetric
Nitrogen	-	-	1-3, 5	inert gas fusion (thermal conductivity)
			4, 6	photometric (nessler reagent)
			7	volumetric (hydrochloric acid)

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).

This certification is applicable to the whole of the disc. However, in accordance with normal practice for OES, it is appropriate to avoid usage of the central portion of ~ 8mm diameter.

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 May 2036, although we reserve the right to make changes as issue revisions, in the intervening period.

This sample is also available in the form of chippings.

The manufacture, analysis and certification of this product were supervised by L Maxim, Technical Director, MBH Analytical Ltd.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.