MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

13X NSA11 (batch A)

Certified Reference Material Information

Type: AUSTENITIC STAINLESS STEEL (WROUGHT)

Form and Size: Disc, ~38mm diameter

Manufactured by: ATI Allvac, SC, USA

Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo
Value [1]	**0.0159**	**0.275**	**<0.001**	**0.0186**	**0.640**	**23.89**	**20.19**	**6.16**
Uncertainty [2]	0.0010	0.008	-	0.0006	0.007	0.10	0.10	0.02

Element	Cu	Co	V	Nb	W	Al	N
Value [1]	**0.187**	**0.0981**	**0.0513**	**0.150**	**0.038**	**(0.021)**	**0.203**
Uncertainty [2]	0.003	0.0016	0.0013	0.003	0.002	-	0.004

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

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 11th March 2013

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial barstock, type AL6XN to UNS N08367. The bar was prepared by electric arc melting, argon-oxygen decarburization and continuous casting, followed by hot forging and annealing.

Sampling

Samples for chemical analysis were taken from various positions throughout the batch. At least 10% of all discs were selected for non-destructive homogeneity testing.

Homogeneity

The discs were checked for lateral segregation, and for local and batch homogeneity 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	0.0140	0.260	0.0002	0.0172	0.623	23.60	19.98	6.141
2	0.0146	0.263	0.0004	0.0172	0.625	23.62	20.00	6.149
3	0.0150	0.265	0.0004	0.0175	0.631	23.72	20.00	6.153
4	0.0150	0.267	0.0005	0.0180	0.631	23.83	20.06	6.156
5	0.0151	0.269	<0.0005	0.0182	0.635	23.86	20.07	6.160
6	0.0152	0.269	<0.0005	0.0182	0.641	23.87	20.12	6.160
7	0.0153	0.271	0.0008	0.0186	0.645	23.88	20.14	6.166
8	0.0160	0.279	0.0010	0.0190	0.645	23.89	20.15	6.168
9	0.0161	0.280	0.0011	0.0191	0.647	23.99	20.25	6.200
10	0.0162	0.292	0.0011	0.0192	0.648	23.99	20.26	
11	0.0165	0.292		0.0195	0.655	24.00	20.27	
12	0.0167	0.293		0.0195	0.656	24.02	20.36	
13	0.0173			0.0196		24.09	20.36	
14	0.0180			0.0198		24.11	20.36	
15	0.0180						20.40	
Mean	**0.0159**	**0.275**	**<0.001**	**0.0186**	**0.640**	**23.89**	**20.19**	**6.161**
Std Dev	0.0012	0.012	-	0.0009	0.011	0.16	0.15	0.017
C (95%)	0.0007	0.008	-	0.0005	0.007	0.09	0.08	0.013

Sample	Cu	Co	V	Nb	W	Al	N
1	0.180	0.0946	0.0486	0.141	0.0346	0.0168	0.191
2	0.181	0.0950	0.0500	0.145	0.0351	0.0180	0.195
3	0.182	0.0952	0.0500	0.145	0.0354	0.0184	0.196
4	0.185	0.0956	0.0500	0.145	0.0366	0.0192	0.197
5	0.187	0.0978	0.0515	0.150	0.0377	0.0193	0.201
6	0.188	0.0985	0.0520	0.150	0.0379	0.0210	0.204
7	0.189	0.0988	0.0520	0.151	0.0381	0.0231	0.205
8	0.189	0.0989	0.0521	0.151	0.0384	0.0242	0.207
9	0.190	0.0996	0.0526	0.152	0.0393	0.0253	0.208
10	0.190	0.1002	0.0545	0.154	0.0415		0.209
11	0.193	0.1014		0.156	0.0419		0.210
12	0.193	0.1019		0.159	0.0430		0.211
Mean	**0.187**	**0.0981**	**0.0513**	**0.150**	**0.0383**	**0.0206**	**0.203**
Std Dev	0.004	0.0025	0.0017	0.005	0.0027	0.0030	0.007
C (95%)	0.003	0.0016	0.0012	0.003	0.0017	0.0023	0.004

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
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Wu Han Steel	WuHan, Hubei, China	CNAL accreditation 0271
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
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
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Coleshill Laboratories Ltd	Birmingham, England	
London & Scandinavian Met. Co Ltd	Rotherham, England	
LECO Corporation	St Joseph, MI, USA	

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	GD-AES	OTHER	
Carbon	-	-	-	all	combustion (IR or volumetric detection)
Silicon	4, 5, 7	-	11	1, 2, 6, 8, 10, 12	gravimetric (perchloric acid)
				3, 9	photometric (molybdenum blue)
Sulfur	-	-	-	all	combustion (IR or volumetric detection)
Phosphorus	1, 2, 6-9, 12, 13	-	4	3, 10, 11	volumetric (alkalimetric)
				5, 14	photometric (molybdenum blue)
Manganese	1-3, 5, 6, 10	4, 7	11	9, 12	photometric (periodate)
				8	volumetric (ferrous ammonium sulfate)
Nickel	1, 3, 4, 6, 11-13	-	8	7, 9, 10, 14	gravimetric (dimethyl glyoxime)
				2, 5	photometric (dimethyl glyoxime)
Chromium	2, 3, 5, 6, 10, 14, 15	-	13	1, 4, 7-9, 11, 12	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 2, 4-6, 8	9	-	3	photometric (thiocyanate)
				7	gravimetric (α benzoin oxime)
Copper	1-4, 7-9, 11, 12	5, 6	10		
Cobalt	2-6, 9, 11, 12	1, 7, 10	-	8	volumetric (iodine)
Vanadium	1-5, 9, 10	8	7	6	volumetric (ferrous ammonium sulfate)
Niobium	1, 3-8, 12	11	10	2	photometric (chlorosulfophenol)
				9	ICP-MS
Tungsten	2, 4-12	-	-	1	ICP-MS
				3	volumetric (titanium chloride)
Aluminium	3-9	1	2		
Nitrogen	-	-	-	5	photometric (Nessler reagent)
				3, 9	volumetric (hydrochloric acid)
				1, 2, 4, 6-8, 10-12	inert gas fusion (thermal conductivity)

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).

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 approx 6mm 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 March 2033, 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.