MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

12X 15256 (batch Q)

Certified Reference Material Information

Type:	LOW-ALLOY STEEL (WROUGHT)
Form and Size:	Disc ~40mm diameter
Manufactured by:	Instytut Metalurgii Zelaza, Poland
Certified and Supplied by:	MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo
Value [1]	**0.123**	**0.190**	**0.0163**	**0.0125**	**0.492**	**5.33**	**0.362**	**0.0740**
Uncertainty [2]	0.003	0.004	0.0007	0.0008	0.003	0.02	0.004	0.0017

Element	Cu	Co	Sn	Al	W	V	Nb	N
Value [1]	**0.0550**	**0.493**	**0.107**	**0.1300**	**0.101**	**0.619**	**0.0509**	**0.0056**
Uncertainty [2]	0.0008	0.008	0.002	0.0015	0.002	0.006	0.0019	0.0003

Definitions

[1] The assigned 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 24th October 2016

MBH ANALYTICAL LIMITED _____

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Method of Preparation

This reference material was produced by vacuum induction melting and ingot casting, followed by hot forging.

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. 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 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 compositional variation of the batch for each element has been quantified by a programme of non-destructive application testing, described 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: Steels are generally prepared by linishing, grinding 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.

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	Ni	Cr	Mo
1	0.1170	0.1809	0.0146	0.0110	0.4859	5.260	0.3500	0.0699
2	0.1180	0.1820	0.0151	0.0113	0.4873	5.300	0.3511	0.0702
3	0.1190	0.1858	0.0152	0.0116	0.4880	5.302	0.3535	0.0716
4	0.1200	0.1870	0.0155	0.0121	0.4905	5.305	0.3545	0.0723
5	0.1205	0.1885	0.0157	0.0122	0.4910	5.309	0.3551	0.0724
6	0.1210	0.1894	0.0157	0.0125	0.4915	5.310	0.3593	0.0726
7	0.1220	0.1902	0.0158	0.0125	0.4920	5.330	0.3620	0.0741
8	0.1249	0.1930	0.0164	0.0125	0.4930	5.336	0.3627	0.0745
9	0.1250	0.1943	0.0165	0.0125	0.4930	5.337	0.3639	0.0746
10	0.1258	0.1950	0.0167	0.0129	0.4969	5.348	0.3651	0.0747
11	0.1302	0.2003	0.0170	0.0131	0.4973	5.350	0.3660	0.0751
12	0.1320		0.0170	0.0132	0.5004	5.350	0.3677	0.0762
13			0.0175	0.0134		5.389	0.3696	0.0767
14			0.0177	0.0138			0.3700	0.0770
15			0.0180				0.3704	0.0784
16							0.3720	
Mean	**0.1230**	**0.1897**	**0.0163**	**0.0125**	**0.4922**	**5.325**	**0.3621**	**0.0740**
Std Dev	0.0047	0.0058	0.0010	0.0008	0.0043	0.032	0.0073	0.0025
$C_{(95\%)}$	0.0030	0.0039	0.0006	0.0005	0.0027	0.019	0.0039	0.0014

Sample	Cu	Co	Sn	Al	W	V	Nb	N
1	0.0534	0.4783	0.1006	0.1254	0.0990	0.6034	0.0468	0.0051
2	0.0542	0.4799	0.1027	0.1284	0.0993	0.6098	0.0476	0.0053
3	0.0547	0.4810	0.1047	0.1286	0.0994	0.6139	0.0491	0.0054
4	0.0547	0.4840	0.1050	0.1290	0.1002	0.6174	0.0497	0.0057
5	0.0547	0.4840	0.1050	0.1293	0.1012	0.6187	0.0502	0.0058
6	0.0548	0.4881	0.1051	0.1300	0.1016	0.6202	0.0504	0.0058
7	0.0555	0.4942	0.1056	0.1304	0.1020	0.6216	0.0511	0.0060
8	0.0559	0.4943	0.1062	0.1306	0.1020	0.6227	0.0525	
9	0.0561	0.4967	0.1073	0.1310	0.1022	0.6250	0.0538	
10	0.0564	0.4972	0.1088	0.1314	0.1028	0.6260	0.0541	
11		0.4977	0.1090	0.1331	0.1049	0.6260	0.0546	
12		0.5004	0.1095	0.1332				
13		0.5005	0.1099					
14		0.5070	0.1100					
15		0.5100	0.1144					
Mean	**0.0550**	**0.4929**	**0.1069**	**0.1300**	**0.1013**	**0.6186**	**0.0509**	**0.0056**
Std Dev	0.0009	0.0099	0.0034	0.0021	0.0018	0.0072	0.0026	0.0003
$C_{(95\%)}$	0.0007	0.0055	0.0019	0.0014	0.0012	0.0048	0.0018	0.0003

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
AnchorCert Analytical	Birmingham, UK	UKAS	accreditation	0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA	accreditation	0492
Genitest Inc.	Montreal, QC, Canada	PRI	accreditation	123077
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			
Analyticka Laborator Lithea sro	Brno, Czech Republic			
Coleshill Laboratories Ltd	Coleshill, UK			

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	-	-	1-12	Combustion (infra-red detection)
Silicon	1, 5-7, 10	-	2, 4, 8	Photometric (molybdenum blue)
			3, 9, 11	Gravimetric (perchloric acid)
Sulfur	2, 10	-	1, 3-9, 11-15	Combustion (infra-red detection)
Phosphorus	1-3, 5, 8-12	-	4, 7, 14	Photometric (molybdenum blue)
			6, 13	Volumetric (alkalimetric)
Manganese	2, 6, 7, 9-12	4, 5	1, 3	Photometric (periodate)
			8	Volumetric (arsenite)
Nickel	2, 4, 6, 9-12	1	3, 5, 7	Gravimetric (dimethyl glyoxime)
			8, 13	Photometric (dimethyl glyoxime)
Chromium	2-8, 12-15	1, 9-10	11, 16	Volumetric (ferrous ammonium sulfate)
Molybdenum	1-5, 7, 9-11, 13, 15	6, 14	8, 12	Photometric (thiocyanate)
Copper	2, 4, 5, 7-10	1, 3	6	Photometric (BCO)
Cobalt	1-3, 6-9, 11-13	5, 10, 15	4	Photometric (2β-naphtol)
			14	Photometric (5-Cl-PADAP)
Tin	2-4, 6-15	1, 5		
Aluminium	2, 3, 5, 7, 8, 10-12	1, 6	4, 9	Photometric (chrome azurol S)
Tungsten	1, 2, 4-7, 9-11	8	3	Photometric (thiocyanate)
Vanadium	1-9	11	10	Volumetric (ferrous ammonium sulfate)
Niobium	1-8, 10, 11	-	9	Photometric (chlorosulfophenol)
Nitrogen	-	-	1	Photometric (Nessler reagent)
			2, 4, 6, 7	Inert gas fusion (thermal conductivity)
			3, 5	Kjeldahl (sulfuric 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 October 2036, 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 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.