MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

12X 16604 (batch A)

Certified Reference Material Information

Type:	LOW-ALLOY STEEL (WROUGHT)
Form and Size:	Disc, ~40mm diameter
Manufactured by:	Bohler Edelstahl, Austria
Certified and Supplied by:	MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr
Value [1]	**0.299**	**0.239**	**0.0018**	**0.0064**	**0.444**	**1.892**	**1.912**
Uncertainty [2]	0.004	0.005	0.0001	0.0007	0.007	0.011	0.018

Element	Mo	Cu	Co	V	Al	Sn	N
Value [1]	**0.334**	**0.131**	**0.0366**	**0.0069**	**0.0111**	**0.0060**	**0.0046**
Uncertainty [2]	0.005	0.003	0.0010	0.0008	0.0012	0.0006	0.0005

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 26th June 2018

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial bar type Bohler V145, to werkstoff 1.6604, also known as 30NICRMO8. The steel was EAF melted, ladle-treated and ingot-cast, prior to hot rolling and tempering.

Sampling

Samples for homogeneity checks and milled samples for chemical analysis were taken from multiple positions within the batch. Approximately 5% of all discs were taken for non-destructive homogeneity testing.

Homogeneity

The samples were checked for lateral and batch uniformity using an optical emission spectrometer.

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 mostly operating within the terms of EN ISO/IEC 17025, 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.

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 milling, turning or grinding. 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
1	0.286	0.2240	0.0016	0.0047	0.4260	1.857	1.865
2	0.292	0.2308	0.0016	0.0052	0.4269	1.858	1.888
3	0.292	0.2314	0.0016	0.0052	0.4304	1.873	1.902
4	0.296	0.2330	0.0017	0.0055	0.4372	1.880	1.907
5	0.297	0.2370	0.0019	0.0055	0.4391	1.882	1.908
6	0.298	0.2370	0.0019	0.0057	0.4410	1.890	1.912
7	0.299	0.2373	0.0020	0.0061	0.4426	1.893	1.921
8	0.301	0.2381	0.0020	0.0061	0.4460	1.898	1.922
9	0.303	0.2422	0.0020	0.0068	0.4530	1.898	1.924
10	0.305	0.2430	0.0021	0.0069	0.4530	1.899	1.928
11	0.305	0.2460		0.0075	0.4550	1.901	1.951
12	0.306	0.2510		0.0078	0.4562	1.905	
13	0.306	0.2530		0.0082	0.4567	1.905	
14				0.0088	0.4590	1.913	
15						1.933	
Mean	**0.299**	**0.2388**	**0.0018**	**0.0064**	**0.4444**	**1.892**	**1.912**
Std Dev	0.006	0.0082	0.0002	0.0013	0.0114	0.020	0.022
C (95%)	0.004	0.0050	0.0001	0.0007	0.0066	0.011	0.015

Sample	Mo	Cu	Co	V	Al	Sn	N
1	0.3215	0.1209	0.0351	0.0052	0.0083	0.0046	0.0037
2	0.3233	0.1217	0.0352	0.0056	0.0084	0.0052	0.0039
3	0.3280	0.1230	0.0352	0.0059	0.0104	0.0052	0.0039
4	0.3284	0.1271	0.0353	0.0059	0.0105	0.0054	0.0045
5	0.3308	0.1275	0.0363	0.0061	0.0109	0.0055	0.0045
6	0.3313	0.1300	0.0363	0.0061	0.0111	0.0059	0.0045
7	0.3350	0.1300	0.0363	0.0062	0.0115	0.0062	0.0050
8	0.3350	0.1319	0.0364	0.0067	0.0123	0.0063	0.0052
9	0.3361	0.1326	0.0367	0.0068	0.0125	0.0066	0.0054
10	0.3381	0.1330	0.0373	0.0076	0.0126	0.0066	0.0058
11	0.3390	0.1340	0.0377	0.0080	0.0140	0.0071	
12	0.3410	0.1341	0.0386	0.0087		0.0076	
13	0.3440	0.1360	0.0389	0.0091			
14	0.3450	0.1369		0.0094			
15		0.1371					
16		0.1373					
Mean	**0.3340**	**0.1308**	**0.0366**	**0.0069**	**0.0111**	**0.0060**	**0.0046**
Std Dev	0.0072	0.0054	0.0013	0.0014	0.0017	0.0009	0.0007
C (95%)	0.0042	0.0029	0.0008	0.0008	0.0012	0.0006	0.0005

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 Analytical Services	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PJ accreditation L17-153
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation 0041
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Luo Yang Copper	Luo Yng, He Nan, China	CNAL accreditation 0173
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
INCDMNR-IMNR	Pantelimon, Romania	
Mineral & Metallurgical Laboratories	Bangalore, India	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

Note: to achieve the above 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	-	-	all	combustion (infra-red detection)
Silicon	1, 2, 6-9, 13	-	3, 4	gravimetric (perchloric acid)
			5, 10-12	photometric (molybdenum blue)
Sulfur	10	-	1-9	combustion (infra-red detection)
Phosphorus	1, 6-13	-	2-5	photometric (molybdenum blue)
			14	volumetric (alkalimetric)
Manganese	1, 2, 4, 5, 7, 9, 12, 13	3, 10	6, 8	volumetric (arsenite)
			11, 14	photometric (periodate)
Nickel	1, 3, 6-10, 12	4, 5, 11, 14	2, 13	photometric (dimethyl glyoxime)
			15	gravimetric (dimethyl glyoxime)
Chromium	1, 2, 4, 6, 10	7, 11	3, 5, 8	volumetric (ferrous ammonium sulfate)
			9	photometric (diphenyl carbazide)
Molybdenum	1-7, 9	10, 12	8, 11, 13, 14	photometric (thiocyanate)
Copper	2-5, 8, 9, 12, 14, 16	1, 6, 13, 15	7, 11	photometric (BCO)
			10	volumetric (thiosulfate)
Cobalt	1, 4, 5, 8-12	2, 3, 13	6, 7	photometric (5 Cl-PADAB)
Vanadium	1-3, 5-9, 12-14	4, 10, 11		
Aluminium	1-3, 5, 7-10	11	4	photometric (chrome azurol S)
			6	volumetric (EDTA)
Tin	1, 3, 4, 6-12	2, 5		
Nitrogen	-	-	1-3, 6, 7,9	inert gas fusion (thermal conductivity)
			4, 5, 8, 10	photometric (Nessler reagent)

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, 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 emission spectrometry, it is appropriate to avoid usage of the centre of the disc, ~8 mm 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 June 2038, 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, for the assessment and calibration of ‘wet’ analytical techniques.

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.