MBH ANALYTICAL LTD

HOLLAND HOUSE • QUEENS ROAD • BARNET • EN5 4DJ • ENGLAND • TEL: +44 (0)20 8441 2024 • FAX: +44 (0)20 8449 0810
email: info@mbh.co.uk web: www.mbh.co.uk

CERTIFICATE OF ANALYSIS

13X 43020 (batch A)

Certified Reference Material Information

Type: FREE-MACHINING STAINLESS STEEL (WROUGHT)

Form and Size: Disc ~40mm diameter

Manufactured by: BGH Edelstahlwerke GmbH, Germany

Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo
Value [1]	**0.147**	**0.415**	**0.189**	**0.0246**	**1.439**	**0.517**	**16.07**	**0.226**
Uncertainty [2]	0.002	0.007	0.004	0.0005	0.011	0.008	0.06	0.004

Element	Cu	Co	V	W	Nb	Al	B	N
Value [1]	**0.0687**	**0.0191**	**0.0542**	**0.0108**	**0.0102**	**0.0047**	**(0.0032)**	**0.0212**
Uncertainty [2]	0.0009	0.0012	0.0017	0.0008	0.0008	0.0005	-	0.0008

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 30th June 2017

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced by arc furnace melting and VOD refining, targeting the composition for werkstoff 1.4104, X14CrMoS17. The steel was continuous-cast, forged to final dimension, then quenched and tempered.

Sampling

Milled samples for chemical analysis were taken from several positions within the bar. In addition, at least 5% of all discs were selected for non-destructive 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, 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.

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, 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	S	P	Mn	Ni	Cr	Mo
1	0.1427	0.4071	0.1796	0.0235	1.413	0.4929	15.87	0.2125
2	0.1430	0.4072	0.1808	0.0237	1.418	0.4970	15.89	0.2163
3	0.1440	0.4080	0.1840	0.0238	1.420	0.5000	15.98	0.2211
4	0.1446	0.4090	0.1840	0.0240	1.427	0.5050	16.01	0.2220
5	0.1455	0.4130	0.1870	0.0240	1.430	0.5080	16.01	0.2227
6	0.1460	0.4203	0.1882	0.0242	1.437	0.5104	16.07	0.2240
7	0.1470	0.4225	0.1890	0.0246	1.444	0.5139	16.10	0.2270
8	0.1470	0.4230	0.1890	0.0249	1.446	0.5206	16.11	0.2284
9	0.1470	0.4280	0.1893	0.0251	1.448	0.5262	16.11	0.2303
10	0.1474		0.1920	0.0252	1.456	0.5280	16.12	0.2310
11	0.1484		0.1930	0.0256	1.457	0.5302	16.14	0.2320
12	0.1490		0.1941	0.0258	1.458	0.5310	16.16	0.2327
13	0.1507		0.1950	0.0259	1.459	0.5337	16.21	0.2330
14	0.1525		0.1950			0.5363	16.21	
15			0.1970					
Mean	**0.1468**	**0.4153**	**0.1891**	**0.0246**	**1.439**	**0.5167**	**16.07**	**0.2256**
Std Dev	0.0028	0.0081	0.0053	0.0008	0.016	0.0146	0.11	0.0065
C (95%)	0.0016	0.0063	0.0030	0.0005	0.010	0.0084	0.06	0.0039

Sample	Cu	Co	V	W	Nb	Al	B	N
1	0.0660	0.0160	0.0501	0.0094	0.0084	0.0036	0.0022	0.0196
2	0.0666	0.0169	0.0503	0.0096	0.0086	0.0036	0.0030	0.0197
3	0.0669	0.0176	0.0519	0.0103	0.0094	0.0040	0.0031	0.0200
4	0.0682	0.0177	0.0521	0.0105	0.0094	0.0044	0.0037	0.0208
5	0.0683	0.0181	0.0534	0.0106	0.0095	0.0044	0.0039	0.0209
6	0.0690	0.0182	0.0541	0.0106	0.0096	0.0045		0.0211
7	0.0691	0.0185	0.0543	0.0109	0.0099	0.0048		0.0212
8	0.0692	0.0190	0.0557	0.0110	0.0104	0.0049		0.0216
9	0.0693	0.0201	0.0561	0.0110	0.0105	0.0051		0.0222
10	0.0696	0.0207	0.0563	0.0111	0.0120	0.0051		0.0223
11	0.0697	0.0213	0.0579	0.0120	0.0121	0.0060		0.0238
12	0.0703	0.0215	0.0580	0.0122	0.0122	0.0060		
13	0.0713	0.0227						
Mean	**0.0687**	**0.0191**	**0.0542**	**0.0108**	**0.0102**	**0.0047**	**(0.0032)**	**0.0212**
Std Dev	0.0015	0.0020	0.0027	0.0008	0.0013	0.0008	-	0.0013
C (95%)	0.0009	0.0012	0.0017	0.0005	0.0008	0.0005	-	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
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
Metals Technology (Testing) Ltd.	Sheffield, UK	UKAS accreditation	0963
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
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
Degerfors Laboratorium AB	Degerfors, Sweden	SWEDAC accreditation	1890
Cogne Acciai Speciali S.p.A.	Aosta, Italy		
Mineral & Metallurgical Laboratories	Bangalore, India		
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, 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 (infra-red detection)
Silicon	2, 4, 6	-	1, 3, 9	gravimetric (perchloric acid)
			5, 7, 8	photometric (molybdenum blue)
Sulfur	6, 12	-	1-5, 7-11, 13-15	combustion (infra-red detection)
Phosphorus	1-3, 5, 6, 8, 9, 11, 13	-	4, 7	photometric (molybdenum blue)
			10, 12	volumetric (alkalimetric)
Manganese	2, 4, 6, 8-12	3, 5	1	photometric (periodate)
			7, 13	volumetric (arsenite)
Nickel	1-3, 7-9, 11, 13, 14	4, 6	5, 10	photometric (dimethyl glyoxime)
			12	gravimetric (dimethyl glyoxime)
Chromium	1, 6-9, 11-13	-	2-7, 10, 14	volumetric (ferrous ammonium sulfate)
Molybdenum	1-3, 5, 7-10, 12	6	4, 11, 13	photometric (thiocyanate)
Copper	1, 5-10, 13	3, 4	2, 12	photometric (BCO)
			11	volumetric (thiosulfate)
Cobalt	1, 3, 5-9, 12, 13	2, 11	4	gravimetric (oxide)
			10	photometric (2β-naphthol)
Vanadium	1, 4-8, 10, 12	2, 3	9, 11	volumetric (ferrous ammonium sulfate)
Tungsten	2-4, 6-12	1	5	gravimetric (cinchonine)
Niobium	1, 3-10, 12	-	2	photometric (chlorosulfophenol)
			11	gravimetric
Aluminium	2-4, 6-8, 10-12	1, 5	9	photometric (chrome azurol S)
Boron	1-5	-		
Nitrogen	-	-	1-4, 6-9	inert gas fusion (thermal conductivity)
			5, 10, 11	photometric (Nessler reagent)

Notes

This Certified Reference Material has been 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 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 June 2037, although we reserve the right to make changes as issue revisions, in the intervening period.

The procurement, 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.