12X 10180 D Page 1 of 4 February 2019

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

12X 10180 (batch D)

# **Certified Reference Material Information**

Type: LOW-ALLOY STEEL (WROUGHT)

Form and Size: Disc, 40mm diameter

Manufactured by: Commercial Bar

Certified and Supplied by: MBH Analytical Ltd

# **Assigned Values**

#### Percentage element by weight

Element	С	Si	S	Р	Mn	Ni	Cr
Value <sup>1</sup>	0.179	0.286	0.0259	0.0148	0.807	0.0531	0.0251
Uncertainty <sup>2</sup>	0.003	0.005	0.0012	0.0010	0.006	0.0011	0.0008

Element	Мо	Cu	Со	Al	Sn	As	N
Value <sup>1</sup>	0.0026	0.0663	0.0070	(0.003)	0.0033	0.0068	0.0070
Uncertainty <sup>2</sup>	0.0002	0.0012	0.0005	-	0.0004	0.0005	0.0012

Note: values in parentheses are not certified; they are provided for information only

# **Definitions**

- 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.
- 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:  MBH ANALYTICAL LIMITED		on 11th February 2019
_	C Eveleigh	<del></del>

# **Method of Preparation**

This reference material was produced from commercial bar to UNS G10180. Details of the production process are unknown.

# **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.

# <u>Usage</u>

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 interelement effects

# **Analytical Data**

### Percentage element by weight

Sample	С	Si	S	Р	Mn	Ni	Cr
1	0.1703	0.2698	0.0221	0.0123	0.7920	0.0502	0.0231
2	0.1710	0.2723	0.0234	0.0127	0.7970	0.0503	0.0237
3	0.1741	0.2820	0.0240	0.0136	0.7998	0.0520	0.0237
4	0.1780	0.2832	0.0241	0.0140	0.8035	0.0520	0.0238
5	0.1782	0.2850	0.0248	0.0142	0.8077	0.0521	0.0238
6	0.1797	0.2853	0.0251	0.0145	0.8089	0.0524	0.0242
7	0.1800	0.2858	0.0252	0.0154	0.8090	0.0525	0.0252
8	0.1800	0.2880	0.0254	0.0155	0.8107	0.0535	0.0252
9	0.1802	0.2890	0.0263	0.0157	0.8120	0.0546	0.0254
10	0.1810	0.2938	0.0263	0.0158	0.8134	0.0549	0.0254
11	0.1823	0.2960	0.0267	0.0166	0.8160	0.0550	0.0260
12	0.1860	0.2970	0.0282	0.0174	0.8183	0.0556	0.0264
13	0.1880		0.0286			0.0558	0.0266
14			0.0288				0.0270
15			0.0289				0.0275
Mean	0.1791	0.2856	0.0259	0.0148	0.8074	0.0531	0.0251
Std Dev	0.0051	0.0084	0.0021	0.0015	0.0079	0.0019	0.0014
C <sub>(95%)</sub>	0.0031	0.0052	0.0012	0.0010	0.0050	0.0011	0.0008
Sample	Мо	Cu	Co	Al	Sn	As	N
Sample 1	<b>Mo</b> 0.0020	<b>Cu</b> 0.0620	<b>Co</b> 0.0055	<b>Al</b> 0.0018	<b>Sn</b> 0.0023	<b>As</b> 0.0055	<b>N</b> 0.0056
1 2							
1 2 3	0.0020	0.0620	0.0055	0.0018	0.0023	0.0055	0.0056
1 2 3 4	0.0020 0.0020 0.0022 0.0022	0.0620 0.0635 0.0652 0.0653	0.0055 0.0066 0.0068 0.0068	0.0018 0.0018 0.0019 0.0019	0.0023 0.0024 0.0025 0.0027	0.0055 0.0056 0.0056 0.0057	0.0056 0.0057 0.0057 0.0062
1 2 3 4 5	0.0020 0.0020 0.0022 0.0022 0.0025	0.0620 0.0635 0.0652 0.0653 0.0658	0.0055 0.0066 0.0068 0.0068 0.0068	0.0018 0.0018 0.0019 0.0019 0.0021	0.0023 0.0024 0.0025 0.0027 0.0028	0.0055 0.0056 0.0056 0.0057 0.0061	0.0056 0.0057 0.0057 0.0062 0.0063
1 2 3 4 5	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660	0.0055 0.0066 0.0068 0.0068 0.0068 0.0069	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030	0.0055 0.0056 0.0056 0.0057 0.0061	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070
1 2 3 4 5 6 7	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670	0.0055 0.0066 0.0068 0.0068 0.0068 0.0069	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072
1 2 3 4 5 6 7 8	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0070	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672	0.0055 0.0066 0.0068 0.0068 0.0068 0.0069 0.0069 0.0070	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072
1 2 3 4 5 6 7 8 9	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0026	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0070 0.0071	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8 9 10	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0026 0.0027	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0070 0.0071 0.0071	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8 9 10 11	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0026 0.0027 0.0027	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0070 0.0071 0.0071 0.0072	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036 0.0037 0.0039	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8 9 10 11 12 13	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0027 0.0027 0.0028	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0070 0.0071 0.0071 0.0072 0.0075	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036 0.0037 0.0039 0.0044	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8 9 10 11	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0026 0.0027 0.0027	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0070 0.0071 0.0071 0.0072	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036 0.0037 0.0039	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8 9 10 11 12 13 14	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0027 0.0027 0.0027 0.0028 0.0028	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0071 0.0071 0.0072 0.0075 0.0075	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036 0.0037 0.0039 0.0044	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 Mean	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0027 0.0027 0.0027 0.0028 0.0028 0.0030	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682 0.0682 0.0687	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0071 0.0071 0.0072 0.0075 0.0075 0.0076 0.0080	0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034 0.0034	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036 0.0037 0.0039 0.0044 0.0046	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093 0.0097
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15	0.0020 0.0020 0.0022 0.0022 0.0025 0.0025 0.0026 0.0026 0.0027 0.0027 0.0027 0.0028 0.0028 0.0030 0.0031	0.0620 0.0635 0.0652 0.0653 0.0658 0.0660 0.0670 0.0672 0.0672 0.0678 0.0682 0.0682 0.0687	0.0055 0.0066 0.0068 0.0068 0.0069 0.0069 0.0071 0.0071 0.0072 0.0075 0.0075 0.0076 0.0080	0.0018 0.0018 0.0019 0.0019 0.0021 0.0023 0.0031 0.0033 0.0034 0.0035	0.0023 0.0024 0.0025 0.0027 0.0028 0.0030 0.0032 0.0033 0.0036 0.0036 0.0037 0.0039 0.0044 0.0046	0.0055 0.0056 0.0056 0.0057 0.0061 0.0064 0.0067 0.0067	0.0056 0.0057 0.0057 0.0062 0.0063 0.0070 0.0072 0.0093 0.0097

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**

Element Ltd Sheffield Analytical Services Metals Technology (Testing) Itd Anchorcert Analytical Universal Scientific Laboratory Pty Ltd Genitest, Inc Shanghai Jinyi Test Tech Co Luo Yang Copper Raghavendra SpectroMet Laboratory TCR Engineering Services Ltd Gesra Labs India Pvt Instytut Metalurgii Zelaza Tec-Eurolab **TUV Nord Czech** INCDMNR-IMNR Mineral & Metallurgical Laboratories AMG Superalloys UK Ltd Analyticka Laborator Lithea sro

Middlesbrough, England Sheffield, England Sheffield, England Birmingham, England Milperra, NSW, Australia Montreal, Canada Shanghai, China Luo Yang, He Nan, China Bangalore, India Mumbai. India Chennai, India Gliwice, Poland Campogalliano, Italy Brno, Czech Republic Pantelimon. Romania Bangalore, India Rotherham, England Brno, Czech Republic

UKAS accreditation 0239
UKAS accreditation 0012
UKAS accreditation 0963
UKAS accreditation 0667
NATA accreditation 0492
PJ accreditation L17-153
CNAS accreditation 0041
CNAL accreditation 0371
NABL accreditation 0367
NABL accreditation 0367
NABL accreditation AB554
ACCREDIA accreditation 52
CAI accreditation L1060

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 (IR or volumetric detection)		
Silicon	1, 2, 4-8, 10	-	3, 9	gravimetric (perchloric acid)		
			5, 11, 12	photometric (molybdenum blue)		
Sulfur	4, 11	-	1-3, 5-10, 12-15	combustion (IR or volumetric detection)		
Phosphorus	1, 3, 4, 7-10	-	2, 11, 12	volumetric (alkalimetric)		
			5, 6	photometric (molybdenum blue)		
Manganese	3-6, 8, 10-12	2	1	photometric (periodate)		
			7, 9	volumetric (arsenite)		
Nickel	1-3, 5, 6, 8-11, 13	4	7, 12	photometric (dimethyl glyoxime)		
Chromium	2, 3, 5-8, 10-12, 14, 15	1, 4, 9	13	volumetric (ferrous ammonium sulfate)		
Molybdenum	2, 3, 6-11, 13-15	5	1, 4, 12	photometric (thiocyanate)		
Copper	1, 5-10, 12, 13	2, 4, 11	3	photometric (BCO)		
Cobalt	1, 4-8, 10, 12-15	3, 9, 11	2	gravimetric		
Aluminium	2-4, 6-11	1, 5				
Tin	1-6, 8, 10-14	7, 9				
Arsenic	1-3, 5, 6, 8, 9	4, 7				
Nitrogen	-	-	1, 3-7, 9	inert gas fusion (thermal conductivity)		
			2, 8	photometric (Nessler reagent)		

#### **Notes**

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO 17034 and the associated Guides, 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 approximately 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 February 2039, 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.