

CERTIFICATE OF ANALYSIS

13X 90901 (batch A)

Certified Reference Material Information

Type: FERRITIC STAINLESS STEEL (WROUGHT)

Form and Size: Disc, ~40mm diameter

Manufactured by: BGH Edelstahl 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	0.1020	0.429	0.0009	0.0148	0.447	0.249	8.43	0.905
Uncertainty ²	0.0015	0.007	0.0001	0.0005	0.005	0.004	0.03	0.006

Element	Cu	V	W	Nb	Al	Ti	Sn	N
Value ¹	0.0399	0.208	0.0094	0.0704	0.0216	(0.0025)	0.0020	0.0472
Uncertainty ²	0.0012	0.003	0.0010	0.0013	0.0017	-	0.0003	0.0009

Note: values 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:

MBH ANALYTICAL LIMITED _____

on 26th February 2019

C EVELEIGH

Method of Preparation

This reference material was produced from commercial barstock to Werkstoff 1.4903, corresponding to ASTM F91, composition UNS K90901. The steel was prepared by electric arc melting and vacuum-oxygen decarburization. The detail casting and bar preparation procedures are unknown.

Sampling

Samples for chemical analysis were taken from various positions throughout the batch. Approximately 5% 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, 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 finishing, 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	0.0988	0.4140	0.00070	0.0135	0.4335	0.2404	8.366	0.8919
2	0.0989	0.4180	0.00078	0.0139	0.4336	0.2411	8.382	0.8977
3	0.0990	0.4185	0.00078	0.0140	0.4386	0.2430	8.382	0.9000
4	0.1001	0.4236	0.00080	0.0141	0.4390	0.2440	8.390	0.9005
5	0.1003	0.4280	0.00080	0.0142	0.4430	0.2440	8.409	0.9010
6	0.1015	0.4327	0.00090	0.0144	0.4442	0.2460	8.422	0.9011
7	0.1019	0.4330	0.00090	0.0145	0.4456	0.2469	8.426	0.9060
8	0.1020	0.4330	0.00090	0.0147	0.4463	0.2480	8.431	0.9064
9	0.1020	0.4339	0.00097	0.0149	0.4466	0.2499	8.436	0.9080
10	0.1020	0.4380	0.00100	0.0150	0.4470	0.2529	8.444	0.9150
11	0.1020	0.4463	0.00108	0.0154	0.4484	0.2551	8.444	0.9161
12	0.1024		0.00115	0.0157	0.4541	0.2570	8.450	0.9219
13	0.1025			0.0163	0.4550	0.2583	8.452	
14	0.1060			0.0164	0.4558	0.2590	8.488	
15	0.1060				0.4633		8.490	
16	0.1070				0.4651			
Mean	0.1020	0.4290	0.00090	0.0148	0.4474	0.2490	8.427	0.9054
Std Dev	0.0025	0.0097	0.00013	0.0009	0.0093	0.0064	0.037	0.0086
C (95%)	0.0013	0.0065	0.00009	0.0005	0.0050	0.0037	0.020	0.0055

Sample	Cu	V	W	Nb	Al	Ti	Sn	N
1	0.0368	0.1978	0.0073	0.0679	0.0182	0.0010	0.0015	0.0444
2	0.0374	0.1990	0.0076	0.0682	0.0184	0.0012	0.0015	0.0460
3	0.0385	0.1997	0.0081	0.0690	0.0188	0.0014	0.0016	0.0464
4	0.0386	0.2030	0.0081	0.0691	0.0190	0.0021	0.0017	0.0468
5	0.0398	0.2047	0.0082	0.0696	0.0198	0.0021	0.0019	0.0470
6	0.0398	0.2067	0.0084	0.0712	0.0214	0.0021	0.0020	0.0472
7	0.0399	0.2080	0.0084	0.0712	0.0224	0.0025	0.0022	0.0473
8	0.0399	0.2085	0.0085	0.0717	0.0229	0.0028	0.0023	0.0478
9	0.0403	0.2094	0.0090	0.0718	0.0232	0.0029	0.0024	0.0488
10	0.0403	0.2100	0.0096	0.0719	0.0232	0.0032	0.0025	0.0490
11	0.0404	0.2120	0.0100	0.0733	0.0254	0.0039	0.0026	0.0490
12	0.0406	0.2125	0.0108		0.0262	0.0042		
13	0.0411	0.2129	0.0110					
14	0.0419	0.2135	0.0115					
15	0.0426	0.2160	0.0122					
16		0.2193	0.0123					
Mean	0.0399	0.2083	0.0094	0.0704	0.0216	0.0025	0.0020	0.0472
Std Dev	0.0015	0.0062	0.0017	0.0018	0.0027	0.0010	0.0004	0.0014
C (95%)	0.0009	0.0033	0.0009	0.0012	0.0017	0.0006	0.0003	0.0009

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) Ltd
Anchorcert Analytical
Laboratory Testing, Inc
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
Hatfield, PA, USA
Milperra, NSW, Australia
Montreal, Canada
Shanghai, China
Luo Yng, 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
A2LA accreditation 0117
NATA accreditation 0492
PJ accreditation L17-153
CNAS accreditation 0041
CNAL accreditation 0173
NABL accreditation 0371
NABL accreditation 0367
NABL accreditation 6238
PCA 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, 3, 6, 8, 9, 11	-	2, 5, 10 4, 7	photometric (molybdenum blue) gravimetric (perchloric acid)
Sulfur	9	-	1-8, 10-12	combustion (IR or volumetric detection)
Phosphorus	1-5, 8-11, 14	-	6, 7, 12 13	photometric (molybdenum blue) volumetric (alkalimetric)
Manganese	1-3, 6-8, 11, 13-16	9	4, 12 5, 10	photometric (periodate) volumetric (arsenite)
Nickel	2, 6, 7, 9-14	3	1, 5, 8 4	photometric (dimethyl glyoxime) gravimetric (dimethyl glyoxime)
Chromium	1, 4-7, 9, 10, 15	3	2, 8, 11-14	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 2, 4-6, 8, 11, 12	10	3, 7, 9	photometric (thiocyanate)
Copper	2, 3, 5-11, 13, 15	1, 4, 14	12	photometric (BCO)
Vanadium	1, 3-6, 8-10, 12-14, 16	7, 15	2, 11	volumetric (ferrous ammonium sulfate)
Tungsten	2-11, 13-16	1	12	gravimetric
Niobium	1-5, 8, 10	9, 11	6 7	gravimetric photometric (chlorosulfophenol)
Aluminium	1, 2, 4, 5, 7-9, 11	3, 12	6 10	volumetric (EDTA) photometric (chrome azurol S)
Titanium	1-4, 6-10, 12	-	5, 11	photometric (peroxide)
Tin	2, 3, 5, 7-11	1, 4, 6		
Nitrogen	-	-	1-8, 10 9, 11	inert gas fusion (thermal conductivity) 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.