

CERTIFICATE OF ANALYSIS

13X NSA9 (batch B)

Certified Reference Material Information

Type: NITROGEN STAINLESS STEEL (WROUGHT)

Form and Size: Disc, ~44mm diameter

Manufactured by: BGH Edelstahl, 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	0.0304	0.290	0.0009	0.0237	1.524	5.75	22.39	3.27
Uncertainty ²	0.0015	0.008	0.0002	0.0009	0.011	0.03	0.05	0.03

Element	Cu	Co	V	Nb	W	B	N
Value ¹	0.154	0.0337	0.0607	0.021	0.033	0.0018	0.184
Uncertainty ²	0.003	0.0015	0.0013	0.002	0.002	0.0002	0.005

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 20th March 2019

C Eveleigh

Method of Preparation

This reference material was produced from commercial barstock: duplex steel prepared to ASTM A276 F51, with nominal composition to UNS S31803. The steel was prepared by electric arc melting and vacuum-oxygen decarburization. It was continuous-cast, hot-rolled, solution-treated and cold-finished

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	0.0257	0.2759	0.0006	0.0212	1.489	5.712	22.25	3.204
2	0.0273	0.2760	0.0006	0.0216	1.502	5.719	22.26	3.211
3	0.0276	0.2819	0.0007	0.0220	1.507	5.719	22.29	3.250
4	0.0280	0.2830	0.0007	0.0224	1.511	5.721	22.30	3.252
5	0.0293	0.2831	0.0007	0.0228	1.522	5.740	22.38	3.252
6	0.0304	0.2866	0.0010	0.0232	1.522	5.748	22.38	3.255
7	0.0304	0.2940	0.0010	0.0236	1.525	5.772	22.39	3.270
8	0.0310	0.2957	0.0010	0.0238	1.527	5.777	22.39	3.277
9	0.0315	0.2980	0.0010	0.0239	1.529	5.790	22.40	3.300
10	0.0318	0.3053	0.0011	0.0241	1.538	5.797	22.42	3.300
11	0.0325	0.3095	0.0012	0.0252	1.544	5.799	22.44	3.344
12	0.0328		0.0013	0.0253	1.547		22.49	3.345
13	0.0334			0.0257	1.552		22.54	
14	0.0335			0.0263			22.55	
Mean	0.0304	0.2902	0.0009	0.0237	1.524	5.754	22.39	3.272
Std Dev	0.0025	0.0120	0.0002	0.0016	0.019	0.034	0.09	0.045
C (95%)	0.0014	0.0081	0.0002	0.0009	0.011	0.023	0.05	0.028

Sample	Cu	Co	V	Nb	W	B	N
1	0.1480	0.0303	0.0571	0.0169	0.0298	0.0014	0.1730
2	0.1500	0.0308	0.0582	0.0175	0.0299	0.0015	0.1741
3	0.1506	0.0310	0.0592	0.0192	0.0301	0.0015	0.1793
4	0.1517	0.0314	0.0597	0.0198	0.0307	0.0015	0.1813
5	0.1520	0.0318	0.0599	0.0204	0.0312	0.0016	0.1820
6	0.1540	0.0335	0.0600	0.0206	0.0340	0.0017	0.1870
7	0.1547	0.0343	0.0602	0.0220	0.0341	0.0020	0.1870
8	0.1550	0.0353	0.0603	0.0222	0.0344	0.0020	0.1871
9	0.1551	0.0353	0.0615	0.0225	0.0359	0.0021	0.1880
10	0.1580	0.0355	0.0617	0.0225	0.0367	0.0022	0.1880
11	0.1590	0.0356	0.0625	0.0225			0.1961
12	0.1618	0.0361	0.0626	0.0248			
13		0.0373	0.0633				
14			0.0639				
Mean	0.1542	0.0337	0.0607	0.0209	0.0327	0.0018	0.1839
Std Dev	0.0040	0.0024	0.0020	0.0023	0.0026	0.0003	0.0068
C (95%)	0.0025	0.0014	0.0011	0.0015	0.0019	0.0002	0.0045

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 Assay Office	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Pty Ltd	Sydney, Australia	NATA accreditation 492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Shandong Metallurgical & Science Research	Shandong, Jinan, China	CNAS accreditation 1461
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Genitest Inc	Montreal, Canada	PJ accreditation L17-153
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Institut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
TUV Nord Czech	Brno, Czech Republic	CAI accreditation L-1060
Mineral & Metallurgical Laboratories	Bangalore, India	
Analyticka Laborator Lithea sro	Brno, Czech Republic	
INCDMNR-IMNR	Pantelimon, Romania	
AMG Superalloys UK Ltd	Rotherham, England	

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

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 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 March 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.