

CERTIFICATE OF ANALYSIS

13X 46500 (batch A)

Certified Reference Material Information

Type: PRECIPITATION-HARDENING STEEL (WROUGHT)

Form and Size: Disc, ~32mm diameter

Manufactured by: Carpenter Technology Corp, USA

Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo	Cu
Value ¹	(0.003)	0.075	0.0010	0.0020	0.004	10.91	11.70	0.948	0.0373
Uncertainty ²	-	0.002	0.0002	0.0002	0.001	0.06	0.05	0.012	0.0013

Element	Co	V	Nb	Ti	Al	Sn	B	N
Value ¹	0.0101	0.0049	0.005	1.51	0.069	0.0030	0.0016	0.0031
Uncertainty ²	0.0008	0.0005	0.001	0.03	0.002	0.0004	0.0002	0.0004

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 28th June 2018
C Eveleigh

Method of Preparation

This reference material was produced from commercial barstock type 'Custom 465' to UNS S46500. The steel was prepared by vacuum induction melting and vacuum arc remelting. The detail 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	Cu
1	0.0013	0.0727	0.00070	0.0015	0.0032	10.77	11.60	0.9350	0.0334
2	0.0019	0.0734	0.00073	0.0017	0.0034	10.80	11.62	0.9362	0.0345
3	0.0020	0.0737	0.00080	0.0018	0.0037	10.81	11.64	0.9387	0.0348
4	0.0021	0.0740	0.00080	0.0019	0.0038	10.82	11.65	0.9394	0.0361
5	0.0026	0.0748	0.00093	0.0019	0.0038	10.84	11.65	0.9400	0.0367
6	0.0026	0.0758	0.00101	0.0020	0.0040	10.89	11.68	0.9410	0.0372
7	0.0034	0.0770	0.00107	0.0020	0.0054	10.92	11.68	0.9417	0.0373
8	0.0038	0.0783	0.00113	0.0022		10.94	11.72	0.9430	0.0385
9	0.0043	0.0788	0.00120	0.0023		10.95	11.75	0.9504	0.0386
10	0.0049		0.00120	0.0023		10.99	11.78	0.9532	0.0391
11	0.0050		0.00120			11.01	11.82	0.9650	0.0392
12			0.00125			11.05	11.82	0.9660	0.0395
13						11.06		0.9690	0.0399
Mean	0.0031	0.0754	0.00100	0.0020	0.0039	10.91	11.70	0.9476	0.0373
Std Dev	0.0013	0.0022	0.00020	0.0003	0.0007	0.10	0.08	0.0120	0.0021
C (95%)	0.0009	0.0017	0.00013	0.0002	0.0007	0.06	0.05	0.0073	0.0013

Sample	Co	V	Nb	Ti	Al	Sn	B	N
1	0.0081	0.0040	0.0031	1.447	0.0653	0.0019	0.0011	0.0023
2	0.0082	0.0042	0.0033	1.464	0.0667	0.0021	0.0014	0.0028
3	0.0089	0.0044	0.0042	1.468	0.0674	0.0029	0.0015	0.0030
4	0.0093	0.0046	0.0043	1.480	0.0674	0.0029	0.0015	0.0031
5	0.0093	0.0048	0.0045	1.480	0.0682	0.0029	0.0016	0.0031
6	0.0098	0.0050	0.0058	1.491	0.0688	0.0029	0.0016	0.0033
7	0.0102	0.0050	0.0060	1.494	0.0688	0.0030	0.0016	0.0034
8	0.0105	0.0050	0.0064	1.507	0.0696	0.0031	0.0018	0.0039
9	0.0109	0.0054	0.0066	1.522	0.0713	0.0032	0.0019	
10	0.0111	0.0057	0.0077	1.543	0.0720	0.0034	0.0021	
11	0.0112	0.0063		1.558		0.0043		
12	0.0115			1.560				
13	0.0125			1.569				
Mean	0.0101	0.0049	0.0052	1.506	0.0686	0.0030	0.0016	0.0031
Std Dev	0.0013	0.0007	0.0015	0.040	0.0020	0.0007	0.0003	0.0005
C (95%)	0.0008	0.0005	0.0011	0.024	0.0015	0.0004	0.0002	0.0004

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
Anchorcert Analytical	Birmingham, England	UKAS accreditation 0667
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
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Mineral & Metallurgical Laboratories	Bangalore, India	
INCDMNR-IMNR	Pantelimon, Romania	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

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

Notes

This Certified Reference Material has been produced and certified, wherever possible, 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 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 June 2038, 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.