

CERTIFICATE OF ANALYSIS

12X 4330V (batch A)

Certified Reference Material Information

Type: LOW-ALLOY STEEL TYPE 4330V (WROUGHT)

Form and Size: Disc, ~45mm diameter

Manufactured by: Tata Steel, Rotherham, UK

Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr
Value ¹	0.327	0.303	0.0030	0.0062	0.857	1.99	0.922
Uncertainty ²	0.004	0.004	0.0002	0.0002	0.006	0.03	0.004

Element	Mo	Cu	Al	V	Sn	As	N
Value ¹	0.470	0.0954	0.0311	0.0507	0.0071	0.0059	0.0078
Uncertainty ²	0.004	0.0012	0.0005	0.0008	0.0004	0.0003	0.0003

Definitions

- ¹ 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.
- 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 15th March 2016

C Eveleigh



Method of Preparation

This reference material was produced from stock commercial bar to ASTM A646 4330.mod 5 (aka 4330V), with compositional specification to UNS K23080. The steel was EAF melted, vacuum-degassed and continuous-cast as bloom, then hot-rolled to final dimension, hardened and tempered.

Sampling

Samples for homogeneity checks and milled samples for chemical analysis were taken from multiple positions within the batch.

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 - 2005, 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.

Usage

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 inter-element effects.

Analytical Data

Percentage element by weight

Sample	C	Si	S	P	Mn	Ni	Cr
1	0.3210	0.2950	0.0025	0.0056	0.8490	1.9432	0.9120
2	0.3211	0.2980	0.0025	0.0057	0.8490	1.9530	0.9126
3	0.3216	0.2990	0.0026	0.0058	0.8501	1.9600	0.9189
4	0.3222	0.2994	0.0026	0.0058	0.8503	1.9709	0.9190
5	0.3224	0.3026	0.0029	0.0061	0.8540	1.9789	0.9198
6	0.3250	0.3027	0.0029	0.0061	0.8540	1.9980	0.9200
7	0.3252	0.3032	0.0029	0.0061	0.8563	2.0003	0.9212
8	0.3259	0.3040	0.0030	0.0062	0.8600	2.0020	0.9215
9	0.3275	0.3044	0.0031	0.0063	0.8604	2.0075	0.9220
10	0.3312	0.3050	0.0032	0.0063	0.8612	2.0169	0.9230
11	0.3320	0.3103	0.0033	0.0063	0.8621	2.0205	0.9250
12	0.3320	0.3105	0.0033	0.0063	0.8630	2.0240	0.9270
13	0.3330	0.3128	0.0034	0.0065	0.8691		0.9340
14	0.3350		0.0036	0.0066			0.9349
15			0.0038	0.0067			
Mean	0.3268	0.3026	0.0030	0.0062	0.8568	1.9896	0.9222
Std Dev	0.0050	0.0052	0.0004	0.0003	0.0064	0.0276	0.0066
C_(95%)	0.0029	0.0031	0.0002	0.0002	0.0038	0.0175	0.0038

Sample	Mo	Cu	Al	V	Sn	As	N
1	0.4591	0.0934	0.0301	0.0488	0.0063	0.0051	0.0072
2	0.4635	0.0941	0.0302	0.0493	0.0064	0.0053	0.0074
3	0.4644	0.0945	0.0304	0.0496	0.0064	0.0055	0.0076
4	0.4660	0.0947	0.0306	0.0498	0.0067	0.0055	0.0078
5	0.4670	0.0950	0.0307	0.0502	0.0068	0.0055	0.0078
6	0.4702	0.0951	0.0308	0.0502	0.0071	0.0056	0.0079
7	0.4708	0.0951	0.0309	0.0507	0.0071	0.0057	0.0080
8	0.4710	0.0952	0.0309	0.0508	0.0071	0.0058	0.0083
9	0.4710	0.0953	0.0310	0.0509	0.0074	0.0058	
10	0.4715	0.0955	0.0313	0.0512	0.0074	0.0059	
11	0.4715	0.0957	0.0315	0.0512	0.0074	0.0060	
12	0.4722	0.0969	0.0321	0.0512	0.0075	0.0065	
13	0.4750	0.0974	0.0326	0.0518	0.0076	0.0066	
14	0.4790	0.0975	0.0329	0.0519	0.0079	0.0071	
15	0.4808			0.0532			
Mean	0.4702	0.0954	0.0311	0.0507	0.0071	0.0059	0.0078
Std Dev	0.0057	0.0012	0.0009	0.0011	0.0005	0.0005	0.0003
C_(95%)	0.0031	0.0007	0.0005	0.0006	0.0003	0.0003	0.0003

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	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Shanghai Jinyi Test Technology Co	Shanghai, China	CNAL accreditation 0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
Bureau Veritas CPS Pvt	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Mineral & Metallurgical Laboratories	Bangalore, India	
Coleshill Laboratories Ltd	Birmingham, England	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea, sro	Brno, Czech Republic	

Note: to achieve the above accreditation (UKAS, NATA, 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, 5-7, 9, 11	-	2, 8, 10 photometric (molybdenum blue)
Sulfur	5, 12	-	3, 4, 12, 13 gravimetric (perchloric acid)
Phosphorus	1, 2, 4-7, 9, 10, 14	-	1-4, 6-11, 13-15 combustion (IR or volumetric detection)
Manganese	3, 4, 6-8, 10, 13	9, 12	3, 8, 13 photometric (molybdenum blue)
Nickel	2, 4, 6, 7, 10, 11	3, 5	11, 12, 15 volumetric (alkalimetric)
Chromium	1, 2, 5, 7, 8, 11, 13, 14	4, 6	1, 2 volumetric (arsenite)
Molybdenum	1-3, 7, 8, 11-15	6, 10	5, 11 photometric (periodate)
Copper	2-6, 10-14	1, 8, 9	1, 12 gravimetric (dimethyl glyoxime)
Aluminium	1, 4-8, 10, 11, 13	2, 9, 12	8, 9 photometric (dimethyl glyoxime)
Vanadium	2, 3, 5, 6, 8, 10, 11, 13-15	1, 7, 12	3, 9, 10 volumetric (ferrous ammonium sulfate)
Tin	1-7, 10, 11, 13, 14	8, 9, 12	12 photometric (diphenyl carbazide)
Arsenic	1-4, 6-11, 13, 14	5, 12	4, 5, 9 photometric (thiocyanate)
Nitrogen	-	-	7 volumetric (thiosulfate)
			13, 14 photometric (BCO)
			3 volumetric (EDTA)
			8, 14 photometric (chrome azurol S)
			4, 9 volumetric (ferrous ammonium sulfate)
			1-3, 7 inert gas fusion (thermal conductivity)
			4, 8 volumetric (hydrochloric acid)
			5, 6 photometric (Nessler reagent)

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2015 and ISO Guide 35-2006, 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 emission spectrometry, it is appropriate to avoid usage of the central portion, ~8 mm 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 testing 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 2036, although we reserve the right to make changes as issue revisions, in the intervening period.

This material is also available in the form of chippings.

The 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.