

CERTIFICATE OF ANALYSIS

13X 14211 (batch R)

Certified Reference Material Information

Type: STAINLESS STEEL (WROUGHT)
Form and Size: Disc, ~40mm diameter
Manufactured by: Instytut Metalurgii Zelaza, Poland
Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo
Value ¹	0.047	1.73	0.0088	0.0093	0.787	12.64	24.48	0.395
Uncertainty ²	0.002	0.02	0.0012	0.0015	0.008	0.07	0.06	0.005

Element	Cu	Co	V	Nb	Ta	W	Ti	Al
Value ¹	0.336	0.034	0.0395	0.150	0.0152	2.99	0.206	0.089
Uncertainty ²	0.007	0.003	0.0013	0.007	0.0003	0.04	0.006	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 uncertainties are value judgements, based on 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 9th November 2019

C Eveleigh

Method of Preparation

This material was produced by vacuum induction melting and ingot casting, followed by hot forging.

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.

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	0.0434	1.704	0.0071	0.0068	0.7720	12.50	24.34	0.3820
2	0.0434	1.706	0.0078	0.0070	0.7743	12.52	24.38	0.3865
3	0.0445	1.709	0.0079	0.0072	0.7780	12.53	24.39	0.3900
4	0.0446	1.709	0.0079	0.0083	0.7800	12.56	24.40	0.3932
5	0.0448	1.712	0.0080	0.0084	0.7830	12.58	24.43	0.3940
6	0.0465	1.714	0.0082	0.0091	0.7860	12.58	24.46	0.3948
7	0.0473	1.720	0.0085	0.0100	0.7870	12.65	24.47	0.3950
8	0.0474	1.740	0.0095	0.0100	0.7894	12.67	24.48	0.3968
9	0.0474	1.761	0.0100	0.0104	0.7905	12.70	24.52	0.3991
10	0.0488	1.771	0.0100	0.0106	0.7926	12.71	24.52	0.4000
11	0.0495	1.779	0.0102	0.0106	0.7998	12.72	24.53	0.4014
12	0.0507		0.0105	0.0108	0.8012	12.73	24.56	0.4020
13				0.0113	0.8030	12.74	24.61	0.4050
14						12.82	24.65	
Mean	0.0465	1.729	0.0088	0.0093	0.7874	12.64	24.48	0.3954
Std Dev	0.0024	0.028	0.0012	0.0016	0.0100	0.10	0.09	0.0065
C (95%)	0.0015	0.019	0.0007	0.0009	0.0060	0.06	0.05	0.0039

Sample	Cu	Co	V	Nb	Ta	W	Ti	Al
1	0.3249	0.0290	0.0362	0.1388	0.0146	2.908	0.1980	0.0832
2	0.3271	0.0304	0.0365	0.1407	0.0148	2.911	0.1990	0.0855
3	0.3287	0.0313	0.0375	0.1430	0.0150	2.937	0.2005	0.0862
4	0.3303	0.0316	0.0378	0.1440	0.0151	2.942	0.2013	0.0868
5	0.3310	0.0329	0.0380	0.1475	0.0152	2.992	0.2017	0.0868
6	0.3310	0.0337	0.0389	0.1480	0.0154	2.994	0.2023	0.0887
7	0.3318	0.0352	0.0391	0.1480	0.0154	2.997	0.2040	0.0907
8	0.3320	0.0366	0.0392	0.1502	0.0155	3.009	0.2050	0.0920
9	0.3350	0.0368	0.0403	0.1553	0.0157	3.010	0.2060	0.0927
10	0.3360	0.0370	0.0406	0.1568		3.020	0.2080	0.0959
11	0.3442	0.0370	0.0408	0.1570		3.033	0.2080	
12	0.3457	0.0373	0.0418	0.1580		3.065	0.2093	
13	0.3500	0.0386	0.0425	0.1597		3.096	0.2110	
14	0.3506		0.0437				0.2178	
15							0.2189	
Mean	0.3356	0.0344	0.0395	0.1498	0.0152	2.993	0.2061	0.0889
Std Dev	0.0085	0.0031	0.0022	0.0070	0.0004	0.057	0.0063	0.0039
C (95%)	0.0049	0.0019	0.0013	0.0042	0.0003	0.034	0.0035	0.0028

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	Middlesbrough, England	UKAS accreditation 0239
Sheffield Analytical Services	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Anchorcert Analytical	Birmingham, England	UKAS accreditation 0667
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PJ accreditation L17-153
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation 0041
Luo Yang Copper	Luo Yng, He Nan, China	CNAL accreditation 0173
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
TUV Nord Czech	Brno, Czech Republic	CAI accreditation L1060
INCDMNR-IMNR	Pantelimon, Romania	
Mineral & Metallurgical Laboratories	Bangalore, India	
AMG Superalloys UK Ltd	Rotherham, England	
LGC Standards	Manchester, NH, USA	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

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, 6, 9, 11	-	2, 3, 8, 10 gravimetric (perchloric acid) 4, 5, 7 photometric (molybdenum blue)
Sulfur	4, 9	-	1-3, 5-8, 10-12 combustion (IR or volumetric detection)
Phosphorus	1-5, 8-10, 12	-	6, 11, 13 photometric (molybdenum blue) 7 volumetric (alkalimetric)
Manganese	1, 2, 6, 8-12	4, 5	3 volumetric (arsenite) 7, 13 photometric (periodate)
Nickel	1, 4-6, 9, 11, 13	-	2, 3, 8, 10, 14 gravimetric (dimethyl glyoxime) 7, 12 photometric (dimethyl glyoxime)
Chromium	2-4, 6, 7, 10, 13, 14	-	1, 5, 8, 9, 11, 12 volumetric (ferrous ammonium sulfate)
Molybdenum	1-4, 7-9, 11, 12	5, 6, 10	13 photometric (thiocyanate)
Copper	1-5, 7, 9, 11, 14	8, 12, 13	6 volumetric (thiosulfate) 10 photometric (BCO)
Cobalt	2, 4, 5, 7-12	1, 3, 13	6 photometric (2β-naphthol)
Vanadium	1-5, 9-13	6, 7	8, 14 volumetric (ferrous ammonium sulfate)
Niobium	1-3, 5, 8-13	6	4 photometric (chlorosulfophenol) 7 gravimetric (N-benzoyl Nph.)
Tantalum	1-9	-	
Tungsten	1, 2, 4-7, 9-11, 13	8, 12	3 photometric (thiocyanate)
Titanium	3-6, 9-12, 14, 15	2, 8, 13	1, 7 photometric (DAP, peroxide)
Aluminium	2-4, 7-10	1, 5	6 photometric (chrome azurol S)

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 centre portion, of diameter ~10mm.

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 November 2039, although we reserve the right to make changes as issue revisions, in the intervening period.

The specification, 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.