MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

12X 12749 (batch X)

Certified Reference Material Information

Type: RESIDUALS IN MILD 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	Cu
Value [1]	**0.176**	**0.480**	**0.066**	**0.0238**	**1.411**	**0.472**	**0.453**	**0.195**	**0.253**
Uncertainty [2]	0.003	0.013	0.003	0.0010	0.015	0.010	0.008	0.006	0.004

Element	Co	V	W	Al	Ti	Sn	As	Pb
Value [1]	**0.426**	**0.0686**	**0.036**	**0.202**	**0.0178**	**0.0188**	**0.081**	**0.0165**
Uncertainty [2]	0.008	0.0011	0.002	0.004	0.0008	0.0012	0.002	0.0013

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:

on 1st August 2019

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This material was produced by vacuum induction melting and ingot casting, followed by hot forging into bar of ~40mm diameter.

Sampling

Samples for wet chemical analysis were taken from several positions within the batch. In addition, approximately 10% of all discs were selected for homogeneity checking.

Homogeneity

Samples representative of the batch were checked for uniformity using an optical emission spectrometer.

For all accepted material, through-batch variation values were derived for each element as an indicator of any minor compositional variation (as determined for the specific sample size and other limitations of 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, using documented standard reference methods and validated by appropriate reference materials.

The individual values listed overpage are the average of each analyst's results.

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.

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.

Usage

Intended use: With optical emission and X-ray fluorescence spectrometers.

Recommended method of use: Steels are generally prepared by linishing, 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.

For optical emission spectroscopy, 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	Mo	Cu
1	0.1695	0.4583	0.0596	0.0216	1.374	0.4579	0.4400	0.1820	0.2410
2	0.1720	0.4650	0.0617	0.0220	1.380	0.4593	0.4420	0.1860	0.2437
3	0.1729	0.4690	0.0619	0.0223	1.390	0.4597	0.4433	0.1874	0.2447
4	0.1730	0.4737	0.0622	0.0225	1.397	0.4598	0.4444	0.1882	0.2480
5	0.1757	0.4750	0.0627	0.0239	1.405	0.4660	0.4454	0.1883	0.2494
6	0.1770	0.4810	0.0642	0.0240	1.408	0.4673	0.4469	0.1900	0.2496
7	0.1770	0.4820	0.0643	0.0240	1.414	0.4705	0.4487	0.1960	0.2500
8	0.1780	0.4896	0.0646	0.0242	1.420	0.4800	0.4490	0.1960	0.2502
9	0.1780	0.4898	0.0670	0.0242	1.430	0.4806	0.4523	0.1962	0.2530
10	0.1783	0.4910	0.0674	0.0255	1.434	0.4830	0.4590	0.1973	0.2542
11	0.1800	0.5080	0.0674	0.0255	1.441	0.4844	0.4647	0.1980	0.2573
12	0.1803		0.0675	0.0261	1.442	0.4846	0.4650	0.2060	0.2590
13			0.0694			0.4873	0.4675	0.2090	0.2600
14			0.0695				0.4680	0.2110	0.2600
15			0.0709						0.2610
16			0.0712						0.2620
Mean	**0.1760**	**0.4802**	**0.0657**	**0.0238**	**1.411**	**0.4723**	**0.4526**	**0.1951**	**0.2527**
Std Dev	0.0034	0.0141	0.0036	0.0015	0.023	0.0113	0.0101	0.0088	0.0066
C (95%)	0.0022	0.0095	0.0019	0.0009	0.015	0.0068	0.0059	0.0051	0.0035

Sample	Co	V	W	Al	Ti	Sn	As	Pb
1	0.4107	0.0649	0.0333	0.1938	0.0155	0.0168	0.0778	0.0145
2	0.4130	0.0671	0.0337	0.1940	0.0173	0.0169	0.0787	0.0146
3	0.4160	0.0672	0.0337	0.1956	0.0173	0.0172	0.0792	0.0148
4	0.4208	0.0678	0.0342	0.1957	0.0174	0.0172	0.0800	0.0149
5	0.4245	0.0683	0.0349	0.1959	0.0177	0.0174	0.0806	0.0158
6	0.4273	0.0683	0.0352	0.1983	0.0177	0.0177	0.0818	0.0158
7	0.4286	0.0688	0.0355	0.2015	0.0177	0.0178	0.0820	0.0158
8	0.4300	0.0692	0.0358	0.2027	0.0178	0.0185	0.0838	0.0168
9	0.4316	0.0697	0.0363	0.2070	0.0182	0.0196	0.0849	0.0169
10	0.4390	0.0697	0.0375	0.2072	0.0190	0.0200	0.0849	0.0183
11	0.4425	0.0698	0.0382	0.2080	0.0199	0.0204		0.0199
12		0.0718	0.0384	0.2080		0.0222		0.0202
13			0.0402	0.2090		0.0225		
14				0.2100				
Mean	**0.4258**	**0.0686**	**0.0359**	**0.2019**	**0.0178**	**0.0188**	**0.0814**	**0.0165**
Std Dev	0.0101	0.0017	0.0021	0.0062	0.0011	0.0020	0.0025	0.0020
C (95%)	0.0068	0.0011	0.0013	0.0036	0.0007	0.0012	0.0018	0.0013

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 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
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
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
TUV Nord-Czech	Brno, Czech Republic	CAI accreditation L-1060
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 (infra-red detection)
Silicon	1-4, 7, 9, 10	-	8, 11	gravimetric (perchloric acid)
			5, 6	photometric (molybdenum blue)
Sulfur	1, 3, 8	-	others	combustion (infra-red detection)
Phosphorus	2-7, 9-11	-	1	photometric (molybdenum blue)
			8, 12	volumetric (alkalimetric)
Manganese	1-8	9	10	photometric (periodate)
			11, 12	volumetric (arsenite, FAS)
Nickel	1-4, 6-8, 11, 13	9, 12	5, 10	photometric (dimethyl glyoxime)
Chromium	1, 3-7, 9-11, 14	2, 12, 13	8	volumetric (ferrous ammonium
Molybdenum	1-11	13	12, 14	photometric (thiocyanate)
Copper	1-3, 5-9, 11, 14, 15	4, 10, 16	12	volumetric thiosulfate)
			13	photometric (BCO)
Cobalt	1, 2, 4-9, 11	3, 10		
Vanadium	2-11	1, 12		
Tungsten	2-11	13	1	gravimetric
			12	photometric (thiocyanate)
Aluminium	1, 3-8, 10, 13, 14	2	9, 11	photometric (chrome azurol S)
			12	volumetric (EDTA)
Titanium	1, 3-9, 11	2	10	photometric (DAP, peroxide)
Tin	1-8, 10- 11, 14, 15	9, 13	12	gravimetric (oxide)
Arsenic	2-10	1		
Lead	1-4, 6-9, 12	10, 11	5	photometric (dithizone)

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 emission spectrometry, it is appropriate to avoid usage of the centre of the disc, ~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 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 August 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 organized and 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.