MBH ANALYTICAL LTD

HOLLAND HOUSE • QUEENS ROAD • BARNET • EN5 4DJ • ENGLAND • TEL: +44 (0)20 8441 2024 • FAX: +44 (0)20 8449 0810

email: info@mbh.co.uk web: www.mbh.co.uk

CERTIFICATE OF ANALYSIS

12X 358 (batch A)

Certified Reference Material Information

Type:	LOW-ALLOY 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]	**0.129**	**0.199**	**0.0142**	**0.0102**	**0.709**	**0.212**	**0.625**	**0.108**
Uncertainty [2]	0.002	0.005	0.0006	0.0011	0.005	0.005	0.004	0.002

Element	Cu	Co	V	W	Al	Ti	Nb	As
Value [1]	**0.250**	**0.0355**	**0.0261**	**0.123**	**0.0616**	**0.0453**	**0.104**	**0.0393**
Uncertainty [2]	0.004	0.0011	0.0007	0.002	0.0011	0.0007	0.003	0.0016

Element	Zr	Sn	Pb	Sb	Se	Bi	N
Value [1]	**0.0113**	**0.117**	**0.0052**	**0.128**	**0.097**	**0.0102**	**0.0029**
Uncertainty [2]	0.0010	0.002	0.0009	0.005	0.002	0.0005	0.0006

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:

on 31st January 2018

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

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

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. The testing procedure was in accordance with ASTM E826 and the material found acceptable.

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 below.

$$C_{(95\%)} = (t \times SD)/\sqrt{n}$$

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.

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
1	0.1237	0.1853	0.0133	0.0079	0.6956	0.1971	0.6115	0.1034
2	0.1240	0.1940	0.0136	0.0082	0.6992	0.2023	0.6159	0.1039
3	0.1250	0.1950	0.0136	0.0083	0.7030	0.2050	0.6200	0.1046
4	0.1266	0.1951	0.0138	0.0085	0.7067	0.2070	0.6226	0.1050
5	0.1280	0.1969	0.0140	0.0087	0.7076	0.2070	0.6241	0.1053
6	0.1290	0.2010	0.0140	0.0089	0.7080	0.2085	0.6256	0.1065
7	0.1290	0.2018	0.0140	0.0101	0.7083	0.2120	0.6258	0.1097
8	0.1309	0.2020	0.0144	0.0104	0.7124	0.2140	0.6267	0.1102
9	0.1310	0.2089	0.0145	0.0104	0.7130	0.2144	0.6280	0.1110
10	0.1312	0.2090	0.0147	0.0108	0.7135	0.2147	0.6283	0.1112
11	0.1330		0.0148	0.0122	0.7175	0.2151	0.6285	0.1117
12	0.1356		0.0152	0.0126	0.7200	0.2190	0.6303	0.1120
13				0.0127		0.2205	0.6310	0.1123
14				0.0134		0.2244	0.6343	
Mean	**0.1289**	**0.1989**	**0.0142**	**0.0102**	**0.7087**	**0.2115**	**0.6252**	**0.1082**
Std Dev	0.0037	0.0072	0.0006	0.0019	0.0071	0.0075	0.0061	0.0034
C (95%)	0.0023	0.0051	0.0004	0.0011	0.0045	0.0043	0.0035	0.0021

Sample	Cu	Co	V	W	Al	Ti	Nb	As
1	0.2425	0.0325	0.0239	0.1175	0.0587	0.0429	0.0965	0.0373
2	0.2427	0.0330	0.0248	0.1180	0.0593	0.0430	0.0990	0.0381
3	0.2427	0.0334	0.0249	0.1205	0.0598	0.0443	0.1007	0.0385
4	0.2439	0.0334	0.0250	0.1209	0.0602	0.0445	0.1015	0.0386
5	0.2451	0.0339	0.0255	0.1210	0.0602	0.0445	0.1027	0.0388
6	0.2452	0.0348	0.0256	0.1220	0.0605	0.0448	0.1042	0.0392
7	0.2470	0.0349	0.0259	0.1220	0.0610	0.0451	0.1060	0.0393
8	0.2495	0.0356	0.0260	0.1231	0.0614	0.0453	0.1060	0.0394
9	0.2508	0.0363	0.0261	0.1231	0.0614	0.0458	0.1070	0.0396
10	0.2530	0.0365	0.0263	0.1245	0.0615	0.0461	0.1070	0.0403
11	0.2540	0.0366	0.0267	0.1249	0.0623	0.0461	0.1094	0.0410
12	0.2562	0.0369	0.0272	0.1253	0.0632	0.0464	0.1127	0.0411
13	0.2585	0.0373	0.0275	0.1270	0.0644	0.0466		
14	0.2608	0.0386	0.0277	0.1281	0.0647	0.0467		
15	0.2614	0.0387	0.0281		0.0651	0.0469		
Mean	**0.2502**	**0.0355**	**0.0261**	**0.1227**	**0.0616**	**0.0453**	**0.1044**	**0.0393**
Std Dev	0.0068	0.0020	0.0012	0.0031	0.0020	0.0013	0.0046	0.0011
C (95%)	0.0037	0.0011	0.0007	0.0018	0.0011	0.0007	0.0029	0.0007

Sample	Zr	Sn	Pb	Sb	Se	Bi	N
1	0.0086	0.1105	0.0030	0.1176	0.0898	0.0090	0.0020
2	0.0092	0.1114	0.0039	0.1193	0.0902	0.0097	0.0023
3	0.0097	0.1136	0.0040	0.1198	0.0929	0.0097	0.0027
4	0.0107	0.1138	0.0046	0.1204	0.0955	0.0098	0.0029
5	0.0109	0.1140	0.0046	0.1251	0.0967	0.0098	0.0030
6	0.0112	0.1157	0.0052	0.1270	0.0967	0.0101	0.0033
7	0.0113	0.1160	0.0056	0.1290	0.0974	0.0103	0.0041
8	0.0114	0.1180	0.0062	0.1320	0.0976	0.0109	
9	0.0118	0.1190	0.0066	0.1328	0.0999	0.0110	
10	0.0119	0.1190	0.0067	0.1352	0.1001	0.0110	
11	0.0120	0.1197	0.0071	0.1370	0.1025	0.0113	
12	0.0137	0.1200		0.1380	0.1034	0.0113	
13	0.0144	0.1210					
14		0.1230					
Mean	**0.0113**	**0.1168**	**0.0052**	**0.1278**	**0.0969**	**0.0102**	**0.0029**
Std Dev	0.0016	0.0038	0.0013	0.0073	0.0043	0.0007	0.0007
C (95%)	0.0010	0.0022	0.0009	0.0047	0.0028	0.0005	0.0006

Note: For the definition and derivation of $C_{(95\%)}$, see page 2:

Participating Laboratories

Exova Ltd	Middlesbrough, England	UKAS accreditation 0239
Anchorcert Ltd	Birmingham, England	UKAS accreditation 0667
Sheffield Analytical Services	Sheffield, England	UKAS accreditation 0012
Special Testing Ltd	Sheffield, England	UKAS accreditation 0046
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
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
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	
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 (infra-red detection)
Silicon	1, 3-5, 9	-	2, 6, 8	photometric (molybdenum blue)
			7, 10	gravimetric (perchloric acid)
Sulfur	3, 4	-	1, 2, 5-12	combustion (infra-red detection)
Phosphorus	1, 2, 4-8, 11, 13	-	3, 10, 12	photometric (molybdenum blue)
			9, 14	volumetric (alkalimetric)
Manganese	2, 4-8, 10, 12	1, 9	3, 11	photometric (periodate)
Nickel	2, 3, 5, 7, 9-11, 13, 14	1, 4, 6	8, 12	photometric (dimethyl glyoxime)
Chromium	1-8, 10, 14	9, 12	11	photometric (diphenyl carbazide)
			13	volumetric (ferrous ammonium sulfate)
Molybdenum	1-3, 5-8, 10, 11, 13	4	9, 12	photometric (thiocyanate)
Copper	1-4, 6, 8, 9, 12, 13, 15	5, 10, 14	7	photometric (BCO)
			11	volumetric (thiosulfate)
Cobalt	1-3, 7-9, 11, 12, 14, 15	4, 13	6	gravimetric (N-benzoyl PH)
			5, 10	photometric (5-chloro-PADAB)
Vanadium	1, 2, 4, 5, 8-11, 13, 15	6, 12	3, 7, 14	volumetric (ferrous ammonium sulfate)
Tungsten	1-4, 7-9, 11, 12, 14	5	6, 10	photometric (thiocyanate)
			13	gravimetric
Aluminium	1-3, 5, 7-12, 14, 15	4	6, 13	photometric (chrome azurol S)
Titanium	1-3, 5, 7, 8, 10, 13-15	11, 12	4, 6, 9	photometric (di-antipyryl methane)
Niobium	1-6, 10-12	7	8, 9	photometric (chlorosulfophenol)
Arsenic	1, 2, 4-12	3		
Zirconium	1-13	-		
Tin	1-11, 14	12	13	gravimetric (oxide)
Lead	1, 2, 5-11	3, 4		
Antimony	1-11	12		
Selenium	1, 3-7, 9-12	2, 8		
Bismuth	2, 3, 5-12	1, 4		
Nitrogen	-	-	1-3, 6, 7	inert gas fusion (thermal conductivity)
			4, 5	photometric (Nessler reagent)

Notes

This Certified Reference Material has been produced and certified 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 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 January 2038, although we reserve the right to make changes as issue revisions, in the intervening period.
This sample is also available in the form of chippings, for the assessment of 'wet' analytical techniques. The manufacture, 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.