

14X MN2 S Page 1 of 4 July 2019

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CERTIFICATE OF ANALYSIS

14X MN2 (batch S)

Certified Reference Material Information

Type: MANGANESE STEEL (CHILL-CAST)

Form and Size: Disc, ~40mm diameter

Manufactured by: Maybrey Reliance Foundry

Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	С	Si	S	Р	Mn	Ni	Cr	Мо
Value 1	0.800	2.20	0.0080	0.0248	12.39	0.726	0.364	1.081
Uncertainty ²	0.014	0.03	0.0006	0.0012	0.10	0.006	0.005	0.010

Element	Cu	V	Nb	Та	Al	Ti	Sn	N
Value 1	0.0894	0.0896	0.239	(0.006)	0.020	0.0189	0.0150	0.0386
Uncertainty ²	0.0013	0.0013	0.012	-	0.002	0.0010	0.0009	0.0015

Note: values given in parentheses are not certified - they are provided for information only.

Definitions

- 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.
- 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 17 th July 2019
MBH ANALYTICAL LIMITED _		
	C Eveleigh	

Method of Preparation

This reference material was produced from commercial-purity metals and master alloys. The discs are the product of one melt poured into multiple chill moulds with feeding systems designed to ensure sound discs. Approximately 2mm has been removed from the cast faces of the discs to minimise surface effects.

Sampling

Milled samples for 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.

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.

<u>Usage</u>

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	С	Si	S	Р	Mn	Ni	Cr	Мо
1	0.7740	2.142	0.0063	0.0221	12.27	0.7094	0.3470	1.053
2 3	0.7743	2.147	0.0065	0.0228	12.30	0.7124	0.3500	1.057
3	0.7810	2.166	0.0069	0.0229	12.34	0.7158	0.3530	1.064
4	0.7900	2.185	0.0073	0.0232	12.34	0.7223	0.3560	1.070
5	0.7945	2.190	0.0075	0.0233	12.35	0.7230	0.3586	1.075
6	0.7951	2.192	0.0077	0.0236	12.37	0.7240	0.3593	1.080
7	0.7962	2.192	0.0079	0.0241	12.37	0.7268	0.3630	1.082
8	0.7970	2.220	0.0080	0.0254	12.40	0.7280	0.3632	1.086
9	0.8057	2.220	0.0082	0.0256	12.42	0.7297	0.3657	1.095
10	0.8164	2.222	0.0083	0.0263	12.45	0.7312	0.3673	1.096
11	0.8220	2.237	0.0085	0.0266	12.47	0.7337	0.3691	1.096
12	0.8220	2.266	0.0086	0.0269	12.47	0.7340	0.3695	1.097
13	0.8270		0.0089	0.0271	12.49	0.7360	0.3755	1.100
14 45			0.0094	0.0271		0.7373	0.3760	
15 16			0.0098				0.3760	
16							0.3784	
Mean	0.7996	2.198	0.0080	0.0248	12.39	0.7260	0.3642	1.081
Std Dev	0.0180	0.037	0.0010	0.0018	0.07	0.0087	0.0097	0.016
C _(95%)	0.0108	0.023	0.0006	0.0011	0.04	0.0050	0.0052	0.010
Sample	Cu	V	Nb	Та	Al	Ti	Sn	N
1	0.0867	0.0865	0.2179	0.0024	0.0181	0.0171	0.0136	0.0354
2	0.0874	0.0875	0.2180	0.0032	0.0186	0.0172	0.0141	0.0359
3	0.0876	0.0894	0.2290	0.0039	0.0186	0.0181	0.0141	0.0367
4	0.0881	0.0895	0.2294	0.0062	0.0188	0.0183	0.0144	0.0372
5	0.0890	0.0898	0.2300	0.0063	0.0188	0.0183	0.0145	0.0377
6	0.0890	0.0902	0.2309	0.0068	0.0191	0.0187	0.0150	0.0390
7	0.0891	0.0902	0.2343	0.0074	0.0197	0.0187	0.0151	0.0390
8	0.0901	0.0906	0.2370	0.0088	0.0218	0.0193	0.0151	0.0398
9	0.0905	0.0924	0.2392	0.0102	0.0227	0.0194	0.0153	0.0404
10	0.0908		0.2418		0.0236	0.0199	0.0155	0.0408
11	0.0912		0.2487		0.0242	0.0199	0.0155	0.0425
12	0.0938		0.2590			0.0205	0.0157	
13			0.2590			0.0209	0.0167	
Mean	0.0894	0.0896	0.2392	(0.0061)	0.0204	0.0189	0.0150	0.0386
Std Dev	0.0020	0.0017	0.0202	-	0.0023	0.0012	0.0008	0.0022

Note: $C_{(95\%)}$ is the 95% half-width confidence interval derived from the equation: $C_{(95\%)}$ = (t x SD)/ \sqrt{n}

0.0122

0.0013

0.0013

C (95%)

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.

0.0015

0.0007

0.0005

0.0015

Participating Laboratories

Element Ltd Anchorcert Ltd Sheffield Analytical Services Metals Technology (Testing) Itd Universal Scientific Laboratory Pty Ltd Genitest, Inc Shanghai Jinyi Test Tech Co Luo Yang Copper Raghavendra SpectroMet Laboratory TCR Engineering Services Ltd Instytut Metalurgii Zelaza Tec-Eurolab **TUV Nord Czech INCDMNR-IMNR** Mineral & Metallurgical Laboratories AMG Superalloys UK Ltd Scrooby's Laboratory Service Analyticka Laborator Lithea sro

Middlesbrough, England Birmingham, England Sheffield, England Sheffield, England Milperra, NSW, Australia Montreal, Canada Shanghai, China Luo Yang, He Nan, China Bangalore, India Mumbai, India Gliwice, Poland Campogalliano, Italy Brno, Czech Republic Pantelimon, Romania Bangalore, India Rotherham, England Benoni, South Africa Brno, Czech Republic

UKAS accreditation 0239
UKAS accreditation 0667
UKAS accreditation 0012
UKAS accreditation 0963
NATA accreditation 0492
PJ accreditation L17-153
CNAS accreditation L0041
CNAL accreditation 0173
NABL accreditation 0371
NABL accreditation 0367
PCA accreditation AB554
ACCREDIA accreditation 52
CAI accreditation L1060

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	9-12	-	1, 3, 5	photometric (molybdenum blue)			
			2, 4, 6-8	gravimetric (perchloric acid)			
Sulfur	7, 10, 11	-	1-6, 8, 9, 12-15	combustion (IR or volumetric detection)			
Phosphorus	1, 4, 5, 7-10, 12-14	-	2, 3, 6	photometric (molybdenum blue)			
			11	volumetric (alkalimetric)			
Manganese	1-4, 8, 10, 11	9	5, 6, 12, 13	volumetric (arsenite, FAS)			
			7	photometric (periodate)			
Nickel	1-4, 6, 7, 9, 11-14	8, 10	5	volumetric (dimethyl glyoxime)			
Chromium	2, 3, 5, 6, 8-14	4, 15, 16	1, 7	volumetric (ferrous ammonium sulfate)			
Molybdenum	1-7, 9-11	13	8, 12	photometric (thiocyanate)			
Copper	1, 2, 4-11	3, 10, 12					
Vanadium	1, 2, 4, 6-9	5	3	volumetric (ferrous ammonium sulfate)			
Niobium	1, 3, 4, 6-10, 12	5, 13	2	photometric (chlorosulfophenol)			
			11	gravimetric (N-benzoyl, N-PH)			
Tantalum	1-9	-					
Aluminium	2, 3, 5, 6, 8-10	4, 11	1	photometric (chrome azurol S)			
			7	volumetric (EDTA)			
Titanium	1-11	-	12, 13	photometric (peroxide, DAP)			
Tin	2, 3, 5-11, 13	1, 12	4	photometric (phenyl fluorone)			
Nitrogen	-	-	1-7, 9	inert gas fusion (thermal conductivity)			
			8, 10, 11	photometric (Nessler reagent)			

Notes

This Certified Reference Material has been produced and certified 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).

The unidirectional solidification effects associated with this method of chill casting, have led to the formation of inhomogeneous segregates in the rear portion of the disc. The above certification is therefore only applicable from the front face of the disc for a depth of 12mm. Material to the rear of the disc, to a depth of ~3 mm, is not certified.

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

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.