

CERTIFICATE OF ANALYSIS

12X 357 (batch D)

Certified Reference Material Information

Type: LOW-ALLOY 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	C	Si	S	P	Mn	Ni	Cr	Cu
Value ¹	0.312	0.211	0.066	0.0101	0.219	0.188	0.212	0.203
Uncertainty ²	0.008	0.005	0.003	0.0007	0.006	0.005	0.010	0.003

Element	Mo	Co	Sn	Al	W	Ti	V	Pb
Value ¹	0.0253	0.198	0.0145	0.138	0.0213	0.074	0.127	0.040
Uncertainty ²	0.0010	0.003	0.0008	0.006	0.0007	0.002	0.003	0.002

Element	Nb	As	Bi	Se	Sb	B	Zr
Value ¹	0.011	0.0127	0.0024	0.0057	0.018	0.0036	0.0049
Uncertainty ²	0.001	0.0006	0.0002	0.0005	0.002	0.0003	0.0003

Note: ^{1,2}: for definitions, see page 2.

Certified by:

MBH ANALYTICAL LIMITED

C Eveleigh

on 12th November 2019

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

Samples for wet chemical analysis were taken from several positions within the batch. In addition, approximately 8% of all discs were selected for homogeneity checking.

Homogeneity and Uncertainty

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Using the combined data from each surface, standard deviation values were derived for each element.

These values were combined with the 95% half-width confidence intervals ($C_{(95\%)}$) obtained from the wet analysis programme, using the square-root of the summed squares, to derive the final uncertainty values.

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.

Usage

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

Recommended method of use: Steels are generally prepared by 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.

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% half-width confidence interval $C_{(95\%)}$, which is derived from the wet analysis results, in accordance with the following 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.

These values have been modified to incorporate statistical analysis of the homogeneity check data, as described above.

Analytical Data

Percentage element by weight

Sample	C	Si	S	P	Mn	Ni	Cr	Cu
1	0.2960	0.1963	0.0599	0.0087	0.2100	0.1760	0.1970	0.1910
2	0.2960	0.2012	0.0600	0.0091	0.2115	0.1770	0.1990	0.1950
3	0.2972	0.2040	0.0646	0.0092	0.2135	0.1770	0.2022	0.1976
4	0.3005	0.2048	0.0647	0.0095	0.2156	0.1795	0.2045	0.1987
5	0.3040	0.2050	0.0652	0.0096	0.2160	0.1795	0.2067	0.2000
6	0.3140	0.2080	0.0661	0.0098	0.2180	0.1857	0.2080	0.2008
7	0.3143	0.2088	0.0662	0.0101	0.2187	0.1880	0.2080	0.2016
8	0.3177	0.2095	0.0663	0.0101	0.2243	0.1896	0.2170	0.2040
9	0.3180	0.2136	0.0665	0.0102	0.2253	0.1915	0.2189	0.2040
10	0.3210	0.2140	0.0671	0.0108	0.2260	0.1920	0.2193	0.2050
11	0.3220	0.2140	0.0674	0.0111	0.2264	0.1947	0.2220	0.2050
12	0.3300	0.2200	0.0675	0.0113	0.2270	0.1960	0.2221	0.2080
13	0.3310	0.2253	0.0731	0.0118		0.1960	0.2230	0.2090
14		0.2260	0.0741			0.1998	0.2240	0.2090
15						0.2000		0.2120
Mean	0.3124	0.2109	0.0663	0.0101	0.2194	0.1882	0.2123	0.2027
Std Dev	0.0125	0.0090	0.0039	0.0009	0.0062	0.0085	0.0096	0.0057
C (95%)	0.0075	0.0052	0.0023	0.0006	0.0039	0.0047	0.0055	0.0032

Sample	Mo	Co	Sn	Al	W	Ti	V	Pb
1	0.0238	0.1880	0.0126	0.1287	0.0192	0.0691	0.1207	0.0365
2	0.0240	0.1885	0.0128	0.1290	0.0199	0.0705	0.1220	0.0368
3	0.0240	0.1932	0.0135	0.1295	0.0203	0.0707	0.1222	0.0379
4	0.0243	0.1960	0.0139	0.1320	0.0210	0.0731	0.1230	0.0385
5	0.0246	0.1965	0.0140	0.1360	0.0212	0.0731	0.1240	0.0391
6	0.0247	0.1980	0.0143	0.1385	0.0212	0.0740	0.1240	0.0399
7	0.0251	0.1990	0.0143	0.1391	0.0215	0.0744	0.1260	0.0400
8	0.0254	0.1995	0.0145	0.1420	0.0217	0.0746	0.1277	0.0400
9	0.0259	0.2012	0.0149	0.1421	0.0219	0.0754	0.1281	0.0402
10	0.0260	0.2020	0.0149	0.1437	0.0219	0.0755	0.1290	0.0412
11	0.0263	0.2024	0.0151	0.1443	0.0227	0.0760	0.1314	0.0417
12	0.0266	0.2030	0.0157	0.1450	0.0232	0.0763	0.1345	0.0425
13	0.0279	0.2035	0.0159	0.1470		0.0777	0.1347	0.0427
14		0.2040	0.0160			0.0780	0.1350	0.0430
Mean	0.0253	0.1982	0.0145	0.1382	0.0213	0.0742	0.1273	0.0400
Std Dev	0.0012	0.0053	0.0011	0.0066	0.0011	0.0027	0.0050	0.0021
C (95%)	0.0007	0.0030	0.0006	0.0040	0.0007	0.0015	0.0029	0.0012

Sample	Nb	As	Bi	Se	Sb	B	Zr
1	0.0086	0.0111	0.0020	0.0047	0.0141	0.0030	0.0039
2	0.0089	0.0117	0.0020	0.0048	0.0148	0.0030	0.0044
3	0.0091	0.0121	0.0020	0.0052	0.0154	0.0030	0.0044
4	0.0100	0.0121	0.0021	0.0054	0.0160	0.0033	0.0047
5	0.0108	0.0124	0.0022	0.0054	0.0164	0.0034	0.0050
6	0.0109	0.0124	0.0022	0.0058	0.0165	0.0036	0.0050
7	0.0111	0.0125	0.0024	0.0062	0.0180	0.0036	0.0050
8	0.0113	0.0127	0.0025	0.0063	0.0188	0.0036	0.0051
9	0.0114	0.0131	0.0025	0.0064	0.0192	0.0037	0.0051
10	0.0116	0.0134	0.0025	0.0070	0.0194	0.0041	0.0052
11	0.0126	0.0135	0.0029		0.0202	0.0041	0.0052
12		0.0136	0.0032		0.0204	0.0042	0.0052
13		0.0149			0.0221		0.0053
Mean	0.0106	0.0127	0.0024	0.0057	0.0178	0.0036	0.0049
Std Dev	0.0013	0.0010	0.0004	0.0007	0.0024	0.0004	0.0004
C (95%)	0.0009	0.0006	0.0002	0.0005	0.0015	0.0003	0.0003

For the definition and derivation of C_(95%), see page 2

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, 2, 4, 5, 7-9, 11-13	-	3 gravimetric (perchloric acid)
Sulfur	1, 7, 9	-	6, 10, 14 photometric (molybdenum blue)
Phosphorus	1-7, 9, 11, 12	-	others combustion (IR or volumetric detection)
Manganese	2-4, 7-11	6	8, 10, 13 photometric (molybdenum blue)
Nickel	1, 4-11, 14, 15	2, 3	1, 5, 12 photometric (periodate)
Chromium	1-5, 8-10, 12, 13	6, 7	12 gravimetric (dimethyl glyoxime)
Copper	1-4, 6-10, 13, 14	5, 11	13 photometric (dimethyl glyoxime)
Molybdenum	1-6, 9-12	7, 8	13 volumetric (ferrous ammonium sulfate)
Cobalt	1-3, 5, 8-14	4, 6	13 photometric (BCO)
Tin	1-5, 7-12, 14	6, 13	13 photometric (thiocyanate)
Aluminium	1, 3, 5-11, 13	2, 12	7 photometric (2β naphthol)
Tungsten	1-5, 7-12	6	4 photometric (chrome azurol S)
Titanium	1-6, 8, 10, 11, 13, 14	9	7, 12 photometric (diantipyryl methane)
Vanadium	1, 3-5, 8-14	6, 7	2 volumetric (ferrous ammonium sulfate)
Lead	1, 3-12, 14	2	13 gravimetric
Niobium	2, 4-11	3	1 photometric (chlorosulfophenol)
Arsenic	3-13	1, 2	
Bismuth	1-6, 9-12	7, 8	
Selenium	1, 3-5, 7-10	2, 6	
Antimony	1-4, 7-12	5, 6	13 gravimetric
Boron	1-12	-	
Zirconium	1-8, 10-13	9	

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).

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, to 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 November 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.