

# CERTIFICATE OF ANALYSIS

**12X 356 (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	Mo
Value <sup>1</sup>	0.228	0.235	0.0403	0.0576	0.325	0.070	0.246	0.0299
Uncertainty <sup>2</sup>	0.005	0.008	0.0012	0.0017	0.004	0.002	0.004	0.0009

Element	Cu	Co	Sn	Al	W	Ti	V	Nb
Value <sup>1</sup>	0.402	0.110	0.0372	0.031	0.086	0.0160	0.071	0.019
Uncertainty <sup>2</sup>	0.005	0.002	0.0008	0.002	0.002	0.0010	0.002	0.002

Element	As	Pb	Bi	Se	Sb	Zn	B	N
Value <sup>1</sup>	0.0250	0.0344	0.0063	0.010	0.0203	0.0111	(0.002)	0.0107
Uncertainty <sup>2</sup>	0.0013	0.0011	0.0005	0.001	0.0009	0.0010	-	0.0006

Note: values in parentheses are not certified; they are provided for information only

## Definitions

- <sup>1</sup> 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.
- <sup>2</sup> 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:

MBH ANALYTICAL LIMITED \_\_\_\_\_  
C Eveleigh

on 5<sup>th</sup> July 2019

## **Method of Preparation**

This reference material was produced from commercial low-carbon steel, with the minor and trace elements added as pure elements or 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 15% 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**

The uncertainty values are generated from 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.

As a separate exercise, the degree of compositional variation of the batch for each element has been quantified by a programme of non-destructive application testing, described above. These values have been combined, using the square-root of the summed squares, to derive the final uncertainty values.

## **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.

## Analytical Data

### Percentage element by weight

Sample	C	Si	S	P	Mn	Ni	Cr	Mo
1	0.2150	0.2150	0.0375	0.0544	0.3161	0.0655	0.2352	0.0271
2	0.2180	0.2230	0.0386	0.0556	0.3178	0.0671	0.2382	0.0291
3	0.2190	0.2250	0.0389	0.0556	0.3181	0.0677	0.2390	0.0293
4	0.2203	0.2283	0.0390	0.0563	0.3210	0.0686	0.2392	0.0295
5	0.2260	0.2290	0.0390	0.0564	0.3220	0.0692	0.2410	0.0295
6	0.2299	0.2324	0.0391	0.0566	0.3240	0.0705	0.2437	0.0298
7	0.2317	0.2343	0.0397	0.0567	0.3251	0.0710	0.2450	0.0299
8	0.2320	0.2387	0.0400	0.0579	0.3255	0.0714	0.2453	0.0299
9	0.2350	0.2390	0.0400	0.0586	0.3260	0.0721	0.2460	0.0303
10	0.2350	0.2458	0.0409	0.0588	0.3270	0.0724	0.2470	0.0304
11	0.2367	0.2530	0.0411	0.0594	0.3285	0.0727	0.2500	0.0307
12	0.2370	0.2576	0.0420	0.0600	0.3290	0.0729	0.2510	0.0308
13			0.0422	0.0625	0.3290	0.0735	0.2515	0.0320
14			0.0422		0.3301		0.2547	
15			0.0423		0.3303		0.2580	
16			0.0428					
<b>Mean</b>	<b>0.2280</b>	<b>0.2351</b>	<b>0.0403</b>	<b>0.0576</b>	<b>0.3246</b>	<b>0.0704</b>	<b>0.2457</b>	<b>0.0299</b>
<b>Std Dev</b>	0.0080	0.0125	0.0016	0.0022	0.0047	0.0025	0.0065	0.0011
<b>C (95%)</b>	0.0051	0.0079	0.0009	0.0013	0.0026	0.0015	0.0036	0.0007

Sample	Cu	Co	Sn	Al	W	Ti	V	Nb
1	0.3900	0.1040	0.0355	0.0277	0.0824	0.0128	0.0685	0.0151
2	0.3927	0.1070	0.0361	0.0298	0.0827	0.0144	0.0686	0.0154
3	0.3941	0.1072	0.0365	0.0303	0.0834	0.0146	0.0694	0.0168
4	0.3954	0.1080	0.0371	0.0304	0.0838	0.0151	0.0699	0.0178
5	0.3961	0.1090	0.0371	0.0307	0.0850	0.0154	0.0700	0.0181
6	0.3975	0.1093	0.0372	0.0308	0.0854	0.0157	0.0707	0.0187
7	0.3980	0.1097	0.0372	0.0309	0.0865	0.0158	0.0708	0.0197
8	0.4001	0.1100	0.0373	0.0316	0.0866	0.0159	0.0722	0.0201
9	0.4020	0.1110	0.0376	0.0316	0.0867	0.0160	0.0742	0.0206
10	0.4078	0.1119	0.0378	0.0327	0.0867	0.0161	0.0743	0.0209
11	0.4080	0.1128	0.0378	0.0336	0.0868	0.0166	0.0744	0.0211
12	0.4090	0.1130	0.0384	0.0348	0.0883	0.0166		0.0231
13	0.4113	0.1135	0.0385		0.0891	0.0190		
14	0.4120	0.1140				0.0193		
15	0.4139	0.1150						
<b>Mean</b>	<b>0.4019</b>	<b>0.1104</b>	<b>0.0372</b>	<b>0.0312</b>	<b>0.0856</b>	<b>0.0160</b>	<b>0.0712</b>	<b>0.0190</b>
<b>Std Dev</b>	0.0078	0.0031	0.0008	0.0018	0.0021	0.0017	0.0023	0.0024
<b>C (95%)</b>	0.0043	0.0017	0.0005	0.0012	0.0013	0.0010	0.0015	0.0015

Sample	As	Pb	Bi	Se	Sb	Zn	B	N
1	0.0210	0.0322	0.0048	0.0075	0.0184	0.0082	0.0016	0.0094
2	0.0225	0.0323	0.0049	0.0087	0.0186	0.0092	0.0017	0.0102
3	0.0237	0.0328	0.0061	0.0087	0.0187	0.0094	0.0018	0.0103
4	0.0239	0.0336	0.0062	0.0089	0.0197	0.0095	0.0019	0.0105
5	0.0244	0.0339	0.0062	0.0093	0.0199	0.0101	0.0026	0.0105
6	0.0250	0.0345	0.0066	0.0095	0.0201	0.0104	0.0033	0.0107
7	0.0251	0.0348	0.0067	0.0100	0.0201	0.0116	0.0038	0.0108
8	0.0255	0.0350	0.0067	0.0103	0.0208	0.0117	0.0041	0.0117
9	0.0255	0.0354	0.0068	0.0116	0.0209	0.0118	0.0050	0.0118
10	0.0256	0.0360	0.0068	0.0122	0.0219	0.0124	0.0066	
11	0.0258	0.0361	0.0071	0.0124	0.0221	0.0128		
12	0.0275	0.0366			0.0224	0.0132		
13	0.0290					0.0137		
<b>Mean</b>	<b>0.0250</b>	<b>0.0344</b>	<b>0.0063</b>	<b>0.0099</b>	<b>0.0203</b>	<b>0.0111</b>	<b>(0.0032)</b>	<b>0.0107</b>
<b>Std Dev</b>	0.0020	0.0015	0.0008	0.0016	0.0014	0.0017	-	0.0007
<b>C (95%)</b>	0.0012	0.0009	0.0005	0.0011	0.0009	0.0010	-	0.0006

## Participating Laboratories

Element Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Analytical Services	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	Milperra, NSW, Australia	NATA accreditation 0492
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Luo Yang Copper	Luo Yang, He Nan, China	CNAL accreditation 0173
Genitest, Inc	Montreal, Canada	PJ accreditation L17-153
Raghavendra Spectromet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
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 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	-	-	2, 3, 9, 12 photometric (molybdenum blue)
Sulfur	1, 6, 12, 13	-	11 gravimetric (perchloric acid)
Phosphorus	1-3, 7, 9, 11-13	-	others combustion (IR or volumetric detection)
Manganese	1-4, 7, 8, 10, 11, 13, 14	6, 15	4, 6 volumetric (alkalimetric)
Nickel	1, 3-5, 8-13	2	5, 8, 10 photometric (molybdenum blue)
Chromium	1-8, 10, 13, 14	9, 11	5, 12 volumetric (arsenite)
Molybdenum	1, 2, 4-11, 13	-	9 photometric (periodate)
Copper	1-6, 8, 11, 13, 15	7, 10, 14	6, 7 photometric (dimethyl glyoxime)
Cobalt	3, 5-8, 10-12, 14	2, 4, 13, 15	12, 15 volumetric (ferrous ammonium sulfate)
Tin	1, 2, 5-12	3, 4, 13	3, 12 photometric (thiocyanate)
Aluminium	1, 4, 7, 9-12	2, 8	9 volumetric (thiosulfate)
Tungsten	1-4, 6, 7, 9, 10, 13	5, 12	12 photometric (BCO)
Titanium	1, 2, 4, 5, 7, 9-13	3, 14	1 gravimetric
Vanadium	1-7, 9, 10	8	9 photometric (5 Chloro-PADAB)
Niobium	1-11	-	9 volumetric (EDTA)
Arsenic	1-3, 5, 6, 8-13	4, 7	5, 6 photometric (chrome azurol S)
Lead	3-6, 8-11	1, 2, 7	8 gravimetric
Bismuth	1-6, 9, 10	7, 8, 11	11 photometric (thiocyanate)
Selenium	1-8, 10, 11	9	11 photometric (diantipryl methane)
Antimony	1-5, 7, 9, 10, 12	6, 8	11 volumetric (ferrous ammonium sulfate)
Zinc	2-6, 8-10, 12	1, 7, 11	12 gravimetric
Boron	1-10	-	12 photometric (dithizone)
Nitrogen	-	-	11 volumetric (bromate)
			1, 9 photometric (Nessler reagent)
			2-8 inert gas fusion (thermal conductivity)

## 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 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 ~5 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.