

CERTIFICATE OF ANALYSIS

11X C1 (batch R)

Certified Reference Material Information

Type: CAST IRON (CHILL CAST)
Form and Size: Disc ~40mm diameter
Produced by: Maybrey Reliance Ltd
Certified and supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Cu	Mo
Value ¹	2.91	1.49	0.057	0.102	1.350	0.557	0.250	0.132	0.0278
Uncertainty ²	0.04	0.03	0.003	0.003	0.016	0.006	0.005	0.003	0.0013

Element	Al	Ti	V	Co	Nb	W	Sn	Zr
Value ¹	0.0195	0.112	0.109	0.0694	0.09	0.100	0.0391	0.0030
Uncertainty ²	0.0014	0.002	0.002	0.0015	0.01	0.005	0.0015	0.0006

Element	As	Sb	Pb	Bi	Zn	Se	B	N
Value ¹	0.0141	0.046	0.005	0.011	0.0097	0.0050	0.0357	0.0091
Uncertainty ²	0.0010	0.002	0.001	0.002	0.0012	0.0005	0.0016	0.0004

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:

MBH ANALYTICAL LIMITED _____ on 30th June 2017
C Eveleigh

Method of Preparation

This reference material was produced from commercial pig iron, 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

Milled samples for chemical analysis were taken from random positions within the casting sequence. In addition, approximately 10% of all discs were selected for non-destructive 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 - 2005, 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: Cast irons 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.

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-elemental effects.

Analytical Data

Percentage element by weight

Sample	C	Si	S	P	Mn	Ni	Cr	Cu	Mo
1	2.850	1.432	0.0530	0.0962	1.318	0.5430	0.2377	0.1270	0.0259
2	2.852	1.453	0.0538	0.0974	1.321	0.5452	0.2390	0.1270	0.0260
3	2.877	1.465	0.0556	0.0987	1.327	0.5480	0.2420	0.1284	0.0264
4	2.879	1.470	0.0556	0.0991	1.331	0.5520	0.2426	0.1290	0.0267
5	2.887	1.474	0.0565	0.0994	1.331	0.5530	0.2460	0.1296	0.0268
6	2.890	1.487	0.0566	0.1030	1.341	0.5560	0.2475	0.1310	0.0274
7	2.896	1.494	0.0568	0.1032	1.348	0.5590	0.2482	0.1320	0.0277
8	2.906	1.495	0.0570	0.1033	1.352	0.5599	0.2487	0.1320	0.0278
9	2.930	1.502	0.0573	0.1040	1.372	0.5602	0.2520	0.1320	0.0278
10	2.930	1.505	0.0580	0.1045	1.380	0.5613	0.2540	0.1332	0.0280
11	2.941	1.514	0.0586	0.1056	1.384	0.5645	0.2552	0.1343	0.0288
12	2.960	1.519	0.0589	0.1060	1.390	0.5688	0.2553	0.1370	0.0296
13	2.970	1.526	0.0595	0.1069		0.5720	0.2650	0.1380	0.0297
14			0.0600				0.2660		0.0304
Mean	2.905	1.487	0.0569	0.1021	1.350	0.5571	0.2499	0.1316	0.0278
Std Dev	0.038	0.027	0.0020	0.0035	0.026	0.0088	0.0087	0.0035	0.0014
C (95%)	0.023	0.017	0.0012	0.0021	0.016	0.0053	0.0050	0.0021	0.0008

Sample	Al	Ti	V	Co	Nb	W	Sn	Zr
1	0.0169	0.1100	0.1038	0.0662	0.0854	0.0966	0.0349	0.0021
2	0.0176	0.1101	0.1051	0.0667	0.0884	0.0970	0.0351	0.0023
3	0.0180	0.1120	0.1060	0.0669	0.0891	0.0974	0.0373	0.0025
4	0.0181	0.1120	0.1070	0.0671	0.0892	0.0990	0.0378	0.0027
5	0.0191	0.1122	0.1085	0.0674	0.0901	0.0997	0.0393	0.0031
6	0.0194	0.1130	0.1099	0.0686	0.0904	0.1005	0.0397	0.0033
7	0.0196	0.1132	0.1102	0.0691	0.0918	0.1040	0.0399	0.0034
8	0.0197	0.1137	0.1104	0.0698	0.0921	0.1054	0.0401	0.0038
9	0.0198	0.1140	0.1115	0.0701	0.0980		0.0409	0.0039
10	0.0201	0.1142	0.1120	0.0713	0.0987		0.0411	
11	0.0214		0.1127	0.0726	0.0994		0.0413	
12	0.0215			0.0730			0.0421	
13	0.0218			0.0734				
Mean	0.0195	0.1124	0.1088	0.0694	0.0921	0.1000	0.0391	0.0030
Std Dev	0.0015	0.0015	0.0030	0.0025	0.0046	0.0032	0.0024	0.0006
C (95%)	0.0009	0.0011	0.0020	0.0015	0.0031	0.0027	0.0015	0.0005

Sample	As	Sb	Pb	Bi	Zn	Se	B	N
1	0.0132	0.0405	0.0026	0.0082	0.0084	0.0038	0.0324	0.0080
2	0.0135	0.0406	0.0029	0.0086	0.0086	0.0039	0.0336	0.0086
3	0.0137	0.0420	0.0038	0.0100	0.0088	0.0040	0.0338	0.0088
4	0.0140	0.0439	0.0040	0.0105	0.0093	0.0044	0.0352	0.0088
5	0.0141	0.0441	0.0043	0.0108	0.0098	0.0047	0.0352	0.0090
6	0.0143	0.0455	0.0046	0.0127	0.0104	0.0050	0.0354	0.0094
7	0.0144	0.0457	0.0053	0.0129	0.0108	0.0056	0.0369	0.0095
8	0.0148	0.0463	0.0054		0.0114	0.0057	0.0376	0.0096
9	0.0150	0.0464	0.0060			0.0058	0.0377	0.0098
10		0.0493	0.0060			0.0058	0.0390	
11		0.0511	0.0064			0.0058		
12		0.0518	0.0069			0.0060		
Mean	0.0141	0.0456	0.0049	0.0105	0.0097	0.0050	0.0357	0.0091
Std Dev	0.0006	0.0037	0.0014	0.0018	0.0011	0.0008	0.0021	0.0006
C (95%)	0.0005	0.0024	0.0009	0.0017	0.0009	0.0005	0.0015	0.0004

For the derivation of the 95% confidence interval, C_(95%), see page 2

Participating Laboratories

Exova Ltd.	Middlesbrough, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
Metals Technology (Testing) Ltd.	Sheffield, UK	UKAS accreditation	0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation	0492
Genitest Inc.	Montreal, QC, Canada	PRI accreditation	123077
Shanghai JinYi Test Technology Co. Ltd	Shanghai, China	CNAL accreditation	0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation	1461
TCR Engineering Services PVT. Ltd.	Mumbai, India	NABL accreditation	0367
Raghavendra Spectro Metallurgical Laboratory	Bangalore, India	NABL accreditation	0371
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation	AB554
TEC Eurolab SRL	Modena, Italy	ACCREDIA accreditation	52
Degerfors Laboratorium AB	Degerfors, Sweden	SWEDAC accreditation	1890
INCDMNR-IMNR	Pantelimon, Romania		
Mineral Metallurgical Laboratories	Bangalore, India		
AMG Superalloys UK Ltd	Rotherham, UK		
Analyticka Laborator Lithea sro	Brno, Czech Republic		
Coleshill Laboratories Ltd	Coleshill, UK		

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, 3-5, 11, 13	-	2, 8, 12	photometric (molybdenum blue)
			6, 7, 9, 10	gravimetric (perchloric acid)
Sulfur	10	-	1-9, 11-14	combustion (infra-red detection)
Phosphorus	1-3, 5, 7, 8, 11-13	-	10	volumetric (alkalimetric)
			4, 6, 9	photometric (molybdenum blue)
Manganese	1, 2, 5-7, 9, 12	4, 10	3	photometric (periodate)
			8, 11	volumetric (arsenite)
Nickel	1, 3, 4, 9-13	2, 8	5, 6	photometric (dimethyl glyoxime)
			7	gravimetric (dimethyl glyoxime)
Chromium	1-5, 7, 9-12	6, 8	13, 14	volumetric (ferrous ammonium sulfate)
Copper	1-3, 5, 6, 9-11, 13	12	4	volumetric (thiosulfate)
			7, 8	photometric (BCO)
Molybdenum	2, 3, 5-10, 12, 13	1, 14	4, 11	photometric (thiocyanate)
Aluminium	1-5, 7, 9, 10, 12, 13	8	6, 11	photometric (chrome azurol S)
Titanium	1, 5-8, 10	2, 9	3, 4	photometric (diantipryl methane)
Vanadium	1, 2, 4, 6, 8-112	5, 7	3	volumetric (ferrous ammonium sulfate)
Cobalt	4-6, 8-10, 12, 13	7, 11	1-3	photometric (5-Cl-PADAP)
Niobium	1, 3-7, 9-11	2	8	photometric (chlorosulfophenol)
Tungsten	2, 4-8	1	3	photometric (thiocyanate)
Tin	2, 3, 5, 6, 8-12	1, 4	7	gravimetric
Zirconium	1-9	-		
Arsenic	2-9	1		
Antimony	2-9, 11, 12	1, 10		
Lead	1-7, 10-12	8, 9		
Bismuth	1, 3, 5-7	2, 4		
Zinc	1-4, 6-8	5		
Selenium	1-12	-		
Boron	1-10	-		
Nitrogen	-	-	1, 7	photometric (Nessler reagent)
			2-6, 8	inert gas fusion (thermal conductivity)
			9	volumetric (sulfuric acid)

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).

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 June 2037, 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.