

# CERTIFICATE OF ANALYSIS

**11X C4 (batch R)**

## Certified Reference Material Information

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

## Assigned Values

### Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo	Cu
Value <sup>1</sup>	1.86	2.92	0.098	0.108	0.493	2.02	1.531	0.101	0.345
Uncertainty <sup>2</sup>	0.02	0.04	0.003	0.003	0.010	0.02	0.012	0.002	0.005

Element	Al	Ti	V	Co	Nb	W	Sb	As	Zn
Value <sup>1</sup>	0.040	0.080	0.0208	0.0316	0.0373	0.120	0.015	0.0050	0.0083
Uncertainty <sup>2</sup>	0.003	0.002	0.0012	0.0011	0.0008	0.004	0.002	0.0005	0.0008

Element	Pb	Sn	Bi	Se	B	Zr	Ag	N
Value <sup>1</sup>	0.019	0.0102	0.0144	0.021	0.0086	0.0031	0.008	0.0078
Uncertainty <sup>2</sup>	0.002	0.0007	0.0008	0.002	0.0004	0.0005	0.001	0.0009

## 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 \_\_\_\_\_

on 28th December 2013

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.

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Milled samples for chemical analysis were taken from random positions within the casting sequence. In addition, at least 15% 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.

Using the meaned data from each surface, standard deviation values were derived for each element as an indicator of any non-homogeneity (as determined for the specific sample size taken by 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. In addition, some of the results derived as part of this testing programme have 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.

Separate standard deviation values were also derived for each element, from the homogeneity testing described above. 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.

## **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-element effects.

## Analytical Data

### Percentage element by weight

Sample	C	Si	S	P	Mn	Ni	Cr	Mo	Cu
1	1.806	2.833	0.0926	0.1040	0.477	1.987	1.503	0.0960	0.334
2	1.830	2.891	0.0939	0.1040	0.478	1.991	1.509	0.0970	0.335
3	1.834	2.900	0.0950	0.1064	0.480	2.006	1.521	0.0990	0.340
4	1.845	2.901	0.0977	0.1065	0.483	2.013	1.526	0.1005	0.342
5	1.851	2.920	0.0978	0.1065	0.483	2.016	1.527	0.1019	0.342
6	1.853	2.929	0.0985	0.1070	0.496	2.020	1.532	0.1021	0.342
7	1.860	2.936	0.0986	0.1071	0.497	2.040	1.533	0.1023	0.344
8	1.870	2.944	0.0992	0.1100	0.497	2.057	1.534	0.1030	0.345
9	1.874	2.945	0.1020	0.1120	0.500		1.541	0.1031	0.349
10	1.890	3.011	0.1030	0.1123	0.508		1.548		0.351
11	1.890		0.1030	0.1130	0.511		1.551		0.351
12	1.900				0.511		1.552		0.352
13	1.925								0.353
<b>Mean</b>	<b>1.864</b>	<b>2.921</b>	<b>0.0983</b>	<b>0.1081</b>	<b>0.493</b>	<b>2.016</b>	<b>1.531</b>	<b>0.1005</b>	<b>0.345</b>
<b>Std Dev</b>	0.032	0.046	0.0035	0.0032	0.013	0.023	0.016	0.0026	0.006
<b>C (95%)</b>	0.020	0.033	0.0023	0.0022	0.008	0.020	0.010	0.0020	0.004

Sample	Al	Ti	V	Co	Nb	W	Sb	As	Zn
1	0.0340	0.0753	0.0181	0.0289	0.0357	0.112	0.0122	0.0034	0.0069
2	0.0365	0.0762	0.0182	0.0290	0.0361	0.114	0.0129	0.0040	0.0075
3	0.0366	0.0784	0.0186	0.0293	0.0365	0.116	0.0132	0.0048	0.0075
4	0.0368	0.0785	0.0189	0.0299	0.0371	0.116	0.0147	0.0049	0.0080
5	0.0374	0.0795	0.0200	0.0315	0.0372	0.121	0.0150	0.0051	0.0081
6	0.0396	0.0795	0.0207	0.0315	0.0374	0.122	0.0154	0.0052	0.0083
7	0.0409	0.0798	0.0207	0.0315	0.0376	0.123	0.0159	0.0052	0.0086
8	0.0413	0.0811	0.0215	0.0321	0.0379	0.123	0.0161	0.0053	0.0090
9	0.0435	0.0829	0.0223	0.0323	0.0385	0.124	0.0183	0.0056	0.0091
10	0.0437	0.0830	0.0223	0.0325	0.0386	0.126	0.0184	0.0056	0.0100
11	0.0443	0.0839	0.0225	0.0334		0.128		0.0061	
12	0.0450	0.0854	0.0234	0.0341					
13			0.0236	0.0342					
<b>Mean</b>	<b>0.0400</b>	<b>0.0803</b>	<b>0.0208</b>	<b>0.0316</b>	<b>0.0373</b>	<b>0.120</b>	<b>0.0152</b>	<b>0.0050</b>	<b>0.0083</b>
<b>Std Dev</b>	0.0037	0.0031	0.0019	0.0018	0.0010	0.005	0.0021	0.0008	0.0009
<b>C (95%)</b>	0.0023	0.0020	0.0012	0.0011	0.0007	0.003	0.0015	0.0005	0.0007

Sample	Pb	Sn	Bi	Se	B	Zr	Ag	N
1	0.0170	0.0090	0.0125	0.0175	0.0080	0.0022	0.0063	0.0065
2	0.0174	0.0096	0.0128	0.0181	0.0080	0.0024	0.0066	0.0070
3	0.0176	0.0097	0.0135	0.0189	0.0082	0.0026	0.0067	0.0080
4	0.0177	0.0098	0.0141	0.0190	0.0085	0.0026	0.0068	0.0080
5	0.0178	0.0100	0.0142	0.0195	0.0087	0.0029	0.0070	0.0085
6	0.0180	0.0102	0.0145	0.0203	0.0091	0.0032	0.0071	0.0088
7	0.0181	0.0105	0.0149	0.0212	0.0092	0.0033	0.0091	
8	0.0184	0.0115	0.0154	0.0239	0.0092	0.0034	0.0097	
9	0.0186	0.0116	0.0154	0.0240		0.0037	0.0098	
10	0.0213		0.0156	0.0248		0.0040	0.0103	
11	0.0217		0.0157			0.0043		
<b>Mean</b>	<b>0.0185</b>	<b>0.0102</b>	<b>0.0144</b>	<b>0.0207</b>	<b>0.0086</b>	<b>0.0031</b>	<b>0.0079</b>	<b>0.0078</b>
<b>Std Dev</b>	0.0016	0.0009	0.0011	0.0026	0.0005	0.0007	0.0016	0.0009
<b>C (95%)</b>	0.0010	0.0007	0.0008	0.0019	0.0004	0.0005	0.0011	0.0009

For the derivation of the 95% confidence interval, C<sub>(95%)</sub>, see page 2

## Participating Laboratories

Exova Ltd  
Sheffield Assay Office  
Metals Technology (Testing) Ltd  
Universal Scientific Laboratory Pty Ltd  
Institute of Iron & Steel Technology  
Shandong Metallurgical & Science Research  
Bureau Veritas CPS Pvt  
TCR Engineering Servs Pvt Ltd  
Raghavendra SpectroMet Laboratory  
Genitest Inc  
Tec-Eurolab  
Instytut Metalurgii Zelaza  
London & Scandinavian Met Co  
Coleshill Laboratories Ltd

Middlesbrough, England  
Sheffield, England  
Sheffield, England  
Milperra, NSW, Australia  
Shanghai, China  
Jinan, Shandong, China  
Chennai, India  
Mumbai, India  
Bangalore, India  
Montreal, Canada  
Campogalliano, Italy  
Gliwice, Poland  
Rotherham, England  
Birmingham, England

UKAS accreditation 0239  
UKAS accreditation 0012  
UKAS accreditation 0963  
NATA accreditation 492  
CNAL accreditation 0783  
CNAS accreditation 1461  
NABL accreditation 0025  
NABL accreditation T367  
NABL accreditation T371  
PRI accreditation 123077  
ACCREDIA accreditation 52  
PCA accreditation AB554

Note: to achieve the above accreditation (UKAS, NATA, 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 (IR or volumetric detection)
Silicon	3, 6, 7, 10	-	1, 2, 4, 5, 8, 9	gravimetric (perchloric acid)
			7	photometric (molybdenum blue)
Sulfur	8	-	1-7, 9-11	combustion (IR or volumetric detection)
Phosphorus	2-4, 6, 10, 11	-	1, 8, 9	photometric (molybdenum blue)
			5, 7	volumetric (alkalimetric)
Manganese	1, 2, 4-6, 8, 9	3, 7	12	volumetric (ferrous ammonium sulfate)
			10, 11	photometric (periodate)
Nickel	1, 2, 6, 7	-	4	gravimetric (dimethyl glyoxime)
			3, 5, 8	photometric (dimethyl glyoxime)
Chromium	1, 2, 4-6, 9, 10	3, 7	8, 11, 12	volumetric (ferrous ammonium sulfate)
Molybdenum	2, 3, 5-7, 9	4	1, 8	photometric (thiocyanate)
Copper	5-13	2-4	1	photometric (BCO)
Aluminium	1, 3-6, 8, 10, 11	2, 7, 9	12	photometric (chrome azurol S)
Titanium	1-4, 6, 8, 9, 11	5, 12	7, 10	photometric (DAP)
Vanadium	1-5, 7, 9-13	6, 8		
Cobalt	1, 3-5, 7-9, 12, 13	2, 6, 10	11	volumetric (iodine)
Niobium	1, 3-5, 7-9	2	6	ICP-MS
			10	photometric (chlorosulfophenol)
Tungsten	1-3, 5, 8-11	6	4	photometric (thiocyanate)
			7	gravimetric (cinchonine)
Antimony	1-8	9, 10		
Arsenic	1-4, 7-11	5, 6		
Zinc	1-7, 10	8, 9		
Lead	1-3, 6-11	4, 5		
Tin	1-6, 8, 9	7		
Bismuth	1, 4-11	2, 3		
Selenium	1, 3, 5-10	2, 4		
Boron	1-5, 7, 8	6		
Zirconium	2-9, 11	10	1	ICP-MS
Silver	1-3, 5-7, 9, 10	4, 8		
Nitrogen	-	-	1, 6	volumetric (hydrochloric acid)
			2-5	combustion (thermal conductivity)

## Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 and ISO Guide 35-2006, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The unidirectional solidification effects associated with 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. Material to the rear of the disc, to a depth of ~3mm, 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 December 2033, 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.