MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

11X C5 (batch W)

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 [1]	**2.52**	**2.09**	**0.096**	**0.080**	**0.846**	**1.595**	**1.111**	**0.471**	**1.80**
Uncertainty [2]	0.03	0.03	0.004	0.004	0.012	0.008	0.012	0.006	0.02

Element	Al	Ti	V	Co	Nb	W	Sb	As	Zn
Value [1]	**0.0484**	**0.054**	**0.089**	**0.0558**	**0.0403**	**0.0297**	**0.0265**	**0.0199**	**0.019**
Uncertainty [2]	0.0013	0.002	0.003	0.0010	0.0015	0.0012	0.0014	0.0012	0.002

Element	Pb	Sn	Bi	Se	B	Zr	Te	N
Value [1]	**0.0295**	**0.0371**	**0.0108**	**0.005**	**0.011**	**0.0050**	**0.0010**	**0.0171**
Uncertainty [2]	0.0015	0.0015	0.0007	0.001	0.001	0.0005	0.0002	0.0007

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:

on 1st July 2013

MBH ANALYTICAL LIMITED _____

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, 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	2.455	2.041	0.0885	0.0756	0.825	1.570	1.087	0.463	1.751
2	2.460	2.052	0.0890	0.0765	0.828	1.581	1.089	0.465	1.761
3	2.479	2.064	0.0924	0.0770	0.833	1.587	1.090	0.465	1.765
4	2.480	2.068	0.0950	0.0781	0.846	1.590	1.090	0.468	1.786
5	2.498	2.079	0.0951	0.0783	0.851	1.590	1.100	0.469	1.787
6	2.514	2.080	0.0960	0.0788	0.853	1.592	1.109	0.470	1.790
7	2.530	2.088	0.0965	0.0800	0.854	1.596	1.110	0.471	1.801
8	2.537	2.110	0.0987	0.0816	0.855	1.602	1.112	0.477	1.803
9	2.540	2.130	0.0990	0.0820	0.856	1.605	1.113	0.478	1.803
10	2.542	2.140	0.0990	0.0835	0.863	1.606	1.124	0.484	1.825
11	2.550	2.142	0.0991	0.0839		1.606	1.130		1.829
12	2.550		0.1000	0.0850		1.614	1.138		1.848
13	2.571			0.0852			1.147		1.850
Mean	**2.516**	**2.090**	**0.0957**	**0.0804**	**0.846**	**1.595**	**1.111**	**0.471**	**1.800**
Std Dev	0.038	0.035	0.0039	0.0033	0.013	0.012	0.020	0.007	0.031
C (95%)	0.023	0.024	0.0025	0.0020	0.009	0.008	0.012	0.005	0.019

Sample	Al	Ti	V	Co	Nb	W	Sb	As	Zn
1	0.0446	0.0483	0.0844	0.0544	0.0379	0.0273	0.0232	0.0169	0.0147
2	0.0454	0.0490	0.0848	0.0546	0.0388	0.0276	0.0238	0.0169	0.0163
3	0.0474	0.0515	0.0853	0.0546	0.0389	0.0285	0.0245	0.0174	0.0175
4	0.0477	0.0517	0.0860	0.0547	0.0390	0.0293	0.0253	0.0185	0.0178
5	0.0483	0.0522	0.0872	0.0548	0.0395	0.0293	0.0254	0.0186	0.0183
6	0.0484	0.0522	0.0875	0.0550	0.0401	0.0294	0.0255	0.0190	0.0185
7	0.0490	0.0532	0.0886	0.0555	0.0401	0.0300	0.0260	0.0194	0.0194
8	0.0490	0.0541	0.0890	0.0556	0.0402	0.0304	0.0264	0.0203	0.0199
9	0.0491	0.0546	0.0899	0.0557	0.0405	0.0304	0.0278	0.0205	0.0200
10	0.0501	0.0561	0.0912	0.0572	0.0409	0.0305	0.0281	0.0208	0.0200
11	0.0502	0.0563	0.0926	0.0575	0.0411	0.0308	0.0283	0.0209	0.0220
12	0.0519	0.0565	0.0929	0.0576	0.0434	0.0313	0.0300	0.0209	0.0240
13		0.0583	0.0946	0.0578	0.0435	0.0315	0.0303	0.0220	
14		0.0587	0.0957					0.0230	
Mean	**0.0484**	**0.0538**	**0.0893**	**0.0558**	**0.0403**	**0.0297**	**0.0265**	**0.0199**	**0.0190**
Std Dev	0.0020	0.0032	0.0037	0.0013	0.0017	0.0013	0.0022	0.0018	0.0025
C (95%)	0.0013	0.0018	0.0021	0.0008	0.0010	0.0008	0.0014	0.0010	0.0016

Sample	Pb	Sn	Bi	Se	B	Zr	Te	N
1	0.0273	0.0333	0.0092	0.0044	0.0080	0.0040	0.0008	0.0160
2	0.0274	0.0335	0.0096	0.0047	0.0082	0.0042	0.0008	0.0164
3	0.0278	0.0348	0.0097	0.0049	0.0093	0.0044	0.0009	0.0165
4	0.0281	0.0351	0.0100	0.0050	0.0103	0.0046	0.0009	0.0170
5	0.0282	0.0358	0.0101	0.0052	0.0104	0.0049	0.0010	0.0174
6	0.0283	0.0361	0.0102	0.0062	0.0106	0.0050	0.0011	0.0178
7	0.0284	0.0362	0.0104	0.0066	0.0108	0.0055	0.0012	0.0180
8	0.0290	0.0362	0.0109		0.0110	0.0055	0.0016	0.0180
9	0.0290	0.0372	0.0110		0.0115	0.0056		
10	0.0305	0.0390	0.0116		0.0118	0.0058		
11	0.0306	0.0400	0.0120		0.0119	0.0060		
12	0.0322	0.0405	0.0127		0.0132			
13	0.0326	0.0405	0.0127		0.0135			
14	0.0330	0.0409						
Mean	**0.0295**	**0.0371**	**0.0108**	**0.0053**	**0.0108**	**0.0050**	**0.0010**	**0.0171**
Std Dev	0.0020	0.0026	0.0012	0.0008	0.0017	0.0007	0.0003	0.0008
C (95%)	0.0011	0.0015	0.0007	0.0007	0.0010	0.0005	0.0002	0.0007

For the derivation of the 95% confidence interval, $C_{(95\%)}$, see page 2

Participating Laboratories

Exova Ltd	Middlesbrough, England	UKAS accreditation 0239
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Wu Han Steel	WuHan, Hubei, China	CNAL accreditation 0271
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
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
Coleshill Laboratories Ltd	Birmingham, England	
London & Scandinavian Met Co	Rotherham, England	
Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (eg 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 (infra-red detection)
Silicon	2, 5, 7, 8, 11	-	1, 3, 6, 10	gravimetric (perchloric acid)
			4, 9	photometric (molybdenum blue)
Sulfur	5, 11	-	1-4, 6-10, 12	combustion (infra-red detection)
Phosphorus	4-9, 11, 13	-	1, 2	volumetric (alkalimetric)
			3, 10, 12	photometric (molybdenum blue)
Manganese	2, 3, 6-8, 10	4	1, 9	volumetric (ferrous ammonium sulfate)
			5	photometric (periodate)
Nickel	1-4, 6, 8, 9, 11, 12	10	5, 7	photometric (dimethyl glyoxime)
Chromium	2-4, 6, 8, 10, 12, 13	5, 7	1, 9, 11	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 3-7, 9, 10	2, 8		
Copper	1, 3, 4, 6, 7, 9-13	5, 8	2	photometric (BCO)
Aluminium	1, 2, 5-10, 12	3, 4	11	photometric (chrome azurol S)
Titanium	1-5, 9-11, 13, 14	7, 12	6	ICP-MS
			8	photometric (diantipyryl methane)
Vanadium	1-6, 8, 9, 11-14	7, 10		
Cobalt	1, 3, 4, 6-9, 12, 13	2, 10, 11	5	volumetric (iodine)
Niobium	1-6, 10-13	9	7	photometric (chlorosulfophenol)
			8	ICP-MS
Tungsten	1, 2, 4-12	-	3	ICP-MS
			13	volumetric (titanium chloride)
Antimony	1-7, 11-13	8-10		
Arsenic	1-3, 5-8, 10-14	4, 9		
Zinc	1-3, 5-7, 9-11	4, 8, 12		
Lead	3-12, 14	1, 2, 13		
Tin	1-6, 8-10, 12, 14	7, 11, 13		
Bismuth	1, 2, 5-12	3, 4, 13		
Selenium	1, 2, 4, 5, 7	3, 6		
Boron	1-8, 10-13	9		
Zirconium	1-4, 6-11	-	5	ICP-MS
Tellurium	2, 4-8	1	3	ICP-MS
Nitrogen	-	-	1, 3-7	combustion (thermal conductivity)
			2	volumetric (hydrochloric acid)
			8	photometric (Nessler reagent)

Notes

This Certified Reference Material has been produced and certified 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 July 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.