MBH ANALYTICAL LTD

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CERTIFICATE OF ANALYSIS

12X 353   (batch F)

Certified Reference Material Information

Type:	LOW-ALLOY STEEL (WROUGHT)
Form and Size:	Disc ~40mm diameter
Manufactured 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
Value [1]	0.122	0.154	0.0200	0.0098	0.644	0.219	0.701	0.107
Uncertainty [2]	0.003	0.003	0.0010	0.0005	0.004	0.004	0.006	0.002

Element	Cu	Co	Sn	Al	W	Ti	V	Pb
Value [1]	0.271	0.0261	0.1178	0.093	0.134	0.059	0.0108	0.0195
Uncertainty [2]	0.002	0.0012	0.0015	0.004	0.004	0.003	0.0003	0.0013

Element	Nb	As	Bi	Se	Sb	Zr	B	N
Value [1]	0.060	0.0489	0.0209	0.0164	0.129	(0.013)	0.0013	0.0112
Uncertainty [2]	0.002	0.0012	0.0007	0.0014	0.002	-	0.0002	0.0006

Note: [1, 2] : for definitions, see page 2.

Note: values given in parentheses are not certified - they are provided for information only.

Certified by:

on 29th September 2015

MBH ANALYTICAL LIMITED _____

C Eveleigh

Method of Preparation

This reference material was produced from commercial-purity metals and master alloys. The discs are the product of one melt, cast into 70mm diameter billets and hot worked into bars of ~42mm diameter.

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 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 - 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.

Usage

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

Recommended   Steels are generally prepared by grinding. However, users are recommended to follow the
method of use: 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 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.

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	Mo
1	0.1180	0.1500	0.0185	0.0088	0.636	0.2116	0.682	0.1015
2	0.1187	0.1508	0.0186	0.0089	0.636	0.2130	0.692	0.1028
3	0.1187	0.1510	0.0189	0.0091	0.638	0.2146	0.692	0.1050
4	0.1194	0.1520	0.0194	0.0093	0.640	0.2161	0.696	0.1050
5	0.1198	0.1520	0.0196	0.0093	0.641	0.2170	0.698	0.1050
6	0.1205	0.1532	0.0198	0.0098	0.642	0.2170	0.699	0.1057
7	0.1220	0.1547	0.0198	0.0099	0.647	0.2180	0.700	0.1058
8	0.1221	0.1557	0.0199	0.0101	0.648	0.2180	0.702	0.1078
9	0.1240	0.1568	0.0203	0.0101	0.649	0.2203	0.707	0.1083
10	0.1240	0.1568	0.0203	0.0102	0.651	0.2207	0.711	0.1085
11	0.1240	0.1575	0.0204	0.0104	0.652	0.2212	0.713	0.1089
12	0.1252	0.1580	0.0208	0.0105	0.653	0.2215	0.714	0.1100
13	0.1270	0.1590	0.0210	0.0106		0.2223		0.1110
14	0.1290		0.0216			0.2229		0.1110
15			0.0218			0.2257		
Mean	**0.1223**	**0.1544**	**0.0200**	**0.0098**	**0.644**	**0.2187**	**0.701**	**0.1069**
Std Dev	0.0034	0.0031	0.0010	0.0006	0.006	0.0039	0.010	0.0029
C (95%)	0.0019	0.0019	0.0005	0.0004	0.004	0.0022	0.006	0.0017

Sample	Cu	Co	Sn	Al	W	Ti	V	Pb
1	0.2660	0.0245	0.1146	0.0863	0.1264	0.0565	0.0100	0.0168
2	0.2665	0.0246	0.1153	0.0870	0.1299	0.0567	0.0101	0.0172
3	0.2681	0.0249	0.1157	0.0890	0.1300	0.0571	0.0102	0.0174
4	0.2690	0.0249	0.1160	0.0898	0.1320	0.0588	0.0104	0.0187
5	0.2695	0.0253	0.1171	0.0902	0.1331	0.0590	0.0104	0.0189
6	0.2699	0.0254	0.1175	0.0903	0.1340	0.0595	0.0105	0.0198
7	0.2705	0.0254	0.1180	0.0907	0.1340	0.0598	0.0107	0.0199
8	0.2730	0.0259	0.1186	0.0908	0.1344	0.0600	0.0108	0.0200
9	0.2735	0.0271	0.1187	0.0967	0.1349	0.0601	0.0110	0.0201
10	0.2740	0.0271	0.1189	0.0967	0.1354	0.0601	0.0110	0.0205
11	0.2740	0.0274	0.1200	0.0968	0.1382	0.0603	0.0113	0.0205
12	0.2752	0.0275	0.1207	0.0971	0.1400	0.0604	0.0114	0.0207
13	0.2757	0.0275	0.1207	0.0974	0.1420		0.0115	0.0209
14		0.0277		0.0990			0.0118	0.0217
Mean	**0.2711**	**0.0261**	**0.1178**	**0.0927**	**0.1342**	**0.0590**	**0.0108**	**0.0195**
Std Dev	0.0033	0.0012	0.0020	0.0043	0.0042	0.0015	0.0006	0.0015
C (95%)	0.0020	0.0007	0.0012	0.0025	0.0026	0.0009	0.0003	0.0009

Sample	Nb	As	Bi	Se	Sb	Zr	B	N
1	0.0566	0.0476	0.0197	0.0140	0.1256	0.0118	0.0010	0.0100
2	0.0578	0.0480	0.0198	0.0145	0.1260	0.0121	0.0010	0.0102
3	0.0582	0.0480	0.0201	0.0145	0.1263	0.0134	0.0011	0.0105
4	0.0585	0.0481	0.0201	0.0151	0.1277	0.0136	0.0012	0.0109
5	0.0589	0.0483	0.0202	0.0154	0.1277	0.0139	0.0012	0.0112
6	0.0603	0.0484	0.0207	0.0169	0.1280	0.0142	0.0012	0.0114
7	0.0606	0.0489	0.0207	0.0173	0.1282	0.0150	0.0013	0.0121
8	0.0618	0.0491	0.0210	0.0178	0.1298		0.0015	0.0121
9	0.0629	0.0495	0.0213	0.0182	0.1300		0.0016	0.0122
10	0.0642	0.0500	0.0221	0.0198	0.1300		0.0016	
11	0.0647	0.0501	0.0224		0.1310		0.0016	
12		0.0503	0.0227		0.1317			
13					0.1336			
Mean	**0.0604**	**0.0489**	**0.0209**	**0.0164**	**0.1289**	**0.0134**	**0.0013**	**0.0112**
Std Dev	0.0027	0.0009	0.0010	0.0019	0.0024	0.0011	0.0002	0.0008
C (95%)	0.0018	0.0006	0.0007	0.0014	0.0014	0.0011	0.0002	0.0006

Participating Laboratories

Exova Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Birmingham Assay Office	Birmingham, England	UKAS accreditation 0667
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Shanghai Jinyi Test Technology Co	Shanghai, China	CNAL accreditation 0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Bureau Veritas CPS Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation 0371
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
London & Scandinavian Met Co Ltd	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Analyticka Laborator 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 (IR or volumetric detection)
Silicon	1, 5, 6, 8, 9, 11, 13	-	2, 7, 10	gravimetric (perchloric acid)
			3, 4, 12	photometric (molybdenum blue)
Sulfur	2, 3	-	1, 4-15	combustion (IR or volumetric detection)
Phosphorus	2, 5, 6, 9-11, 13	-	1, 4, 8	volumetric (alkalimetric)
			3, 7, 12	photometric (molybdenum blue)
Manganese	1, 2, 6, 8, 10-12	4, 5	3, 9	photometric (periodate)
			7	volumetric (arsenite)
Nickel	2-4, 6, 8-11, 13-15	1, 12	5, 7	photometric (dimethyl glyoxime)
Chromium	1, 3, 4, 6-9, 12	2	5, 11	volumetric (ferrous ammonium sulfate)
			10	photometric (diphenyl carbazide)
Molybdenum	1-3, 6-8, 10, 11, 13, 14	4, 9	5, 12	photometric (thiocyanate)
Copper	1, 4-9, 12, 13	2, 10	3, 11	photometric (BCO)
Cobalt	1, 3-6, 8, 10, 11, 14	7, 9, 12	2	volumetric (iodine)
			13	photometric (2β naphthol)
Tin	1-3, 5, 7-10, 12, 13	4, 6, 11		
Aluminium	1-5, 7-9, 11, 12	10, 13	6, 14	photometric (chrome azurol S)
Tungsten	1-6, 8, 10-12	13	7	photometric (thiocyanate); 9: ICP-MS
Titanium	1, 4-8, 12	3, 11	2, 10	photometric (diantipyryl methane); 9: ICP-MS
Vanadium	1-4, 6-8, 10-14	5, 9		
Lead	1, 2, 4-10, 12-14	3, 11		
Niobium	1, 2, 4-6, 8, 10, 11	9	7	photometric (chlorosulfophenol); 3: ICP-MS
Arsenic	1-6, 8-10, 12	7, 11		
Bismuth	1-3, 6, 7, 9-12	4, 5, 8		
Selenium	1-8, 10	9		
Antimony	1-6, 8, 9, 11-13	7, 10		
Zirconium	1, 2, 4, 6, 7	3	5	ICP-MS
Boron	1-11	-		
Nitrogen	-	-	2, 5	volumetric (hydrochloric acid)
			4, 7	photometric (Nessler reagent)
			1, 3, 6, 8, 9	inert gas fusion (thermal conductivity detection)

Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2015 and ISO Guide 35-2006, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

This certification is applicable to the whole of the disc. However, in accordance with normal practice for emission spectrometry, it is appropriate to avoid usage of the centre of the disc, ~8 mm diameter.

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 September 2035, 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.