

CERTIFICATE OF ANALYSIS

12X 355 (batch B)

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	0.152	0.486	0.0284	0.0289	0.595	0.081	0.103	0.0603
Uncertainty ²	0.003	0.013	0.0009	0.0013	0.005	0.003	0.002	0.0013

Element	Cu	Co	Sn	Al	W	Ti	V	Nb
Value ¹	0.632	0.0515	0.0547	0.118	0.0294	0.144	0.121	0.039
Uncertainty ²	0.004	0.0013	0.0015	0.002	0.0015	0.003	0.002	0.002

Element	As	Pb	Bi	Se	Sb	Zr	B	N
Value ¹	0.0294	0.0302	0.043	0.0338	0.080	0.087	(0.0010)	0.0067
Uncertainty ²	0.0013	0.0009	0.003	0.0012	0.004	0.005	-	0.0003

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

^{1,2}: for definitions, see page 2.

Certified by:

MBH ANALYTICAL LIMITED _____

on 8th November 2012

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, at least 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 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.

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	0.145	0.462	0.0268	0.0258	0.580	0.0766	0.0985	0.0583
2	0.145	0.463	0.0268	0.0260	0.584	0.0772	0.0985	0.0585
3	0.147	0.463	0.0272	0.0270	0.585	0.0775	0.0997	0.0600
4	0.149	0.483	0.0272	0.0281	0.593	0.0783	0.1007	0.0601
5	0.151	0.489	0.0285	0.0289	0.594	0.0791	0.1013	0.0605
6	0.152	0.495	0.0292	0.0289	0.595	0.0792	0.1027	0.0607
7	0.152	0.495	0.0292	0.0299	0.596	0.0809	0.1028	0.0618
8	0.153	0.501	0.0295	0.0303	0.599	0.0833	0.1032	0.0626
9	0.155	0.501	0.0296	0.0308	0.599	0.0844	0.1040	
10	0.155	0.511	0.0300	0.0310	0.600	0.0854	0.1049	
11	0.157			0.0315	0.605	0.0868	0.1065	
12	0.160				0.609	0.0875	0.1076	
Mean	0.152	0.486	0.0284	0.0289	0.595	0.0814	0.1025	0.0603
Std Dev	0.005	0.018	0.0013	0.0020	0.009	0.0039	0.0029	0.0015
C (95%)	0.003	0.013	0.0009	0.0013	0.005	0.0025	0.0019	0.0012

Sample	Cu	Co	Sn	Al	W	Ti	V	Nb
1	0.623	0.0485	0.0523	0.113	0.0257	0.139	0.116	0.0355
2	0.628	0.0489	0.0525	0.113	0.0265	0.139	0.116	0.0355
3	0.629	0.0493	0.0531	0.115	0.0277	0.140	0.117	0.0370
4	0.629	0.0498	0.0532	0.116	0.0282	0.141	0.118	0.0379
5	0.631	0.0511	0.0533	0.117	0.0287	0.141	0.119	0.0382
6	0.632	0.0515	0.0533	0.118	0.0304	0.142	0.121	0.0384
7	0.634	0.0521	0.0539	0.118	0.0305	0.142	0.121	0.0412
8	0.636	0.0521	0.0549	0.118	0.0307	0.147	0.122	0.0414
9	0.641	0.0531	0.0557	0.120	0.0312	0.148	0.123	0.0427
10	0.641	0.0532	0.0563	0.121	0.0313	0.148	0.124	
11		0.0536	0.0563	0.125	0.0325	0.148	0.124	
12		0.0545	0.0581			0.149	0.129	
13			0.0582			0.151		
Mean	0.632	0.0515	0.0547	0.118	0.0294	0.144	0.121	0.0386
Std Dev	0.006	0.0020	0.0020	0.004	0.0022	0.004	0.004	0.0026
C (95%)	0.004	0.0013	0.0012	0.002	0.0015	0.003	0.002	0.0020

Sample	As	Pb	Bi	Se	Sb	Zr	B	N
1	0.0263	0.0281	0.0405	0.0302	0.0734	0.0773	0.0005	0.0063
2	0.0275	0.0292	0.0408	0.0313	0.0743	0.0807	0.0006	0.0065
3	0.0286	0.0292	0.0409	0.0322	0.0756	0.0838	0.0010	0.0067
4	0.0289	0.0293	0.0415	0.0337	0.0766	0.0847	0.0010	0.0067
5	0.0289	0.0294	0.0421	0.0339	0.0769	0.0848	0.0010	0.0068
6	0.0291	0.0302	0.0422	0.0340	0.0786	0.0851	0.0010	0.0071
7	0.0295	0.0309	0.0422	0.0342	0.0810	0.0883	0.0011	0.0071
8	0.0297	0.0312	0.0441	0.0345	0.0819	0.0887	0.0012	
9	0.0306	0.0320	0.0472	0.0346	0.0827	0.0909	0.0013	
10	0.0321	0.0320	0.0474	0.0350	0.0850	0.0956	0.0014	
11	0.0321		0.0482	0.0357	0.0853	0.0962		
12				0.0368	0.0862			
Mean	0.0294	0.0302	0.0434	0.0338	0.0798	0.0869	0.0010	0.0067
Std Dev	0.0017	0.0013	0.0029	0.0018	0.0045	0.0058	0.0003	0.0003
C (95%)	0.0012	0.0009	0.0019	0.0012	0.0028	0.0039	0.0002	0.0003

Participating Laboratories

Exova Materials Testing Ltd
Metals Technology Testing, Ltd
Sheffield Assay Office
Universal Scientific Laboratory Pty
Laboratory Testing, Inc
Genitest Inc
Institute of Iron & Steel Technology
Sargam Laboratory Pvt Ltd
TCR Engineering Servs Pvt Ltd
Raghavendra SpectroMet Laboratory
Instytut Metalurgii Zelaza
London & Scandinavian Met. Co Ltd
De Bruyn Spectroscopic Solutions
Coleshill Laboratories Ltd

Middlesbrough, England
Sheffield, England
Sheffield, England
Milperra, NSW, Australia
Hatfield, PA, USA
Montreal, Canada
Shanghai, China
Chennai, India
Mumbai, India
Bangalore, India
Gliwice, Poland
Rotherham, England
Johannesburg, South Africa
Birmingham, England

UKAS accreditation 0239
UKAS accreditation 0963
UKAS accreditation 0012
NATA accreditation 0492
A2LA accreditation 0117
PRI accreditation 123077
CNAL accreditation 0783
NABL accreditation T025
NABL accreditation T367
NABL accreditation T371
PCA accreditation AB554

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, 3, 5, 9	-	4	photometric (molybdenum blue)
			2, 6-8, 10	gravimetric (perchloric acid)
Sulfur	5, 6	-	1-4, 7-10	combustion (IR or volumetric detection)
Phosphorus	4-7, 9-11	-	1, 2	volumetric (alkalimetric)
			3, 8	photometric (molybdenum blue)
Manganese	2-5, 7-10	1, 6	11	photometric (periodate)
			12	volumetric (arsenite)
Nickel	1, 3, 5, 6, 8-12	2, 4, 7		
Chromium	1-7, 10-12	8	9	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 2, 5-8	3, 4		
Copper	1-3, 5, 7-9	4, 6, 10		
Cobalt	1, 4-9, 11	3, 10, 12	2	photometric (5-chloro PADAB)
Tin	2-4, 6-11, 13	1, 5, 12		
Aluminium	1-3, 6-11	4, 5		
Tungsten	1-4, 6-8, 10	-	9	volumetric (titanium chloride); 5: ICP-MS
			11	gravimetric (cinchonine)
Titanium	1-3, 6, 7, 10-13	4, 8	9	photometric (diantipyryl methane); 5: ICP-MS
Vanadium	1-3, 5-9, 11, 12	4, 10		
Niobium	2-6, 8, 9	-	7	photometric (chlorosulfophenol); 1: ICP-MS
Arsenic	1, 2, 4-11	3		
Lead	1, 3-5, 7-10	2, 6		
Bismuth	1-4, 6, 7, 10	5, 8, 9	11	ICP-MS
Selenium	1, 2, 5, 6, 8-12	3, 7	4	ICP-MS
Antimony	1, 2, 4-11	3, 12		
Zirconium	1-6, 8-11	-	7	ICP-MS
Boron	1-10	-		
Nitrogen	-	-	5, 6	photometric (Nessler reagent, after distillation)
			1-4, 7	inert gas fusion (thermal conductivity detection)

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).

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. This certification will therefore expire in November 2032, although we reserve the right to make changes as issue revisions, in the intervening period.

This sample is also available in the form of chippings.

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.