

CERTIFICATE OF ANALYSIS

12X 12747 (batch U)

Certified Reference Material Information

Type: LOW-ALLOY STEEL (WROUGHT)
Form and Size: Disc 42mm Diameter x 15mm Thickness
Manufactured by: Polycast Ltd
Certified and Supplied by: MBH Analytical Limited

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo
Value ¹	0.149	0.337	0.041	0.029	2.02	0.391	0.443	0.500
Uncertainty ²	0.003	0.005	0.002	0.002	0.02	0.006	0.013	0.006

Element	Cu	Co	V	W	Al	Ti	Sn	As
Value ¹	0.437	0.200	0.0375	0.030	0.015	0.072	0.167	0.0114
Uncertainty ²	0.007	0.004	0.0015	0.003	0.002	0.002	0.007	0.0005

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 10th October 2007

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 chemical analysis were taken from various positions throughout the casting process. At least 15% of all discs were selected for non-destructive homogeneity testing.

Homogeneity

The discs were checked for sample and batch uniformity using an optical emission spectrometer.

Using the combined 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 operating within the terms of EN ISO/IEC 17025 - 2000, using documented standard reference methods and validated by appropriate reference materials.

The individual values listed overpage are the average of each analyst's results.

Estimation of Uncertainties

Each element certified has been analysed by several laboratories, and 95% half-width confidence intervals ($C_{(95\%)}$) for the resultant mean values have been derived by the method shown on page 3.

As a separate exercise, the degree of non-homogeneity of the batch for each element has been quantified by a programme of non-destructive application testing, discussed above.

The final certified uncertainty for each element has been derived by combining these two factors, using the square-root of the summed squares.

Traceability

Most of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to a national authority. It is part of the 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 primary reference materials.

Usage

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

Recommended method of use: Steels are generally prepared by finishing, grinding, turning or milling. 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.

The recommended sample size is at least five replicate analyses. 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
1	0.142	0.326	0.0376	0.0252	1.980	0.374	0.416	0.487
2	0.145	0.327	0.0378	0.0254	2.00	0.380	0.416	0.489
3	0.146	0.330	0.0383	0.0278	2.004	0.384	0.422	0.490
4	0.147	0.331	0.0386	0.0279	2.009	0.385	0.430	0.494
5	0.147	0.334	0.0390	0.0286	2.014	0.388	0.433	0.498
6	0.148	0.335	0.0394	0.0289	2.020	0.389	0.434	0.500
7	0.148	0.336	0.0406	0.0294	2.024	0.390	0.439	0.502
8	0.148	0.339	0.0410	0.0299	2.027	0.392	0.453	0.504
9	0.150	0.342	0.0420	0.0309	2.050	0.395	0.459	0.505
10	0.152	0.343	0.0422	0.0320	2.051	0.396	0.466	0.509
11	0.155	0.348	0.0429	0.034		0.400	0.471	0.510
12	0.158	0.348	0.0453			0.405	0.475	0.516
13			0.0460			0.406		
Mean	0.149	0.337	0.0408	0.0291	2.018	0.391	0.443	0.500
Std Dev	0.004	0.008	0.0027	0.0026	0.022	0.009	0.021	0.009
C (95%)	0.003	0.005	0.0017	0.0018	0.016	0.006	0.013	0.006

Sample	Cu	Co	V	W	Al	Ti	Sn	As
1	0.422	0.190	0.0349	0.0252	0.0092	0.0684	0.150	0.0106
2	0.424	0.190	0.036	0.0259	0.0115	0.0690	0.154	0.0112
3	0.425	0.197	0.036	0.0263	0.0134	0.0697	0.156	0.0113
4	0.427	0.198	0.036	0.027	0.0135	0.070	0.161	0.0115
5	0.435	0.198	0.0374	0.028	0.014	0.0709	0.167	0.0116
6	0.436	0.198	0.0375	0.0283	0.0143	0.0710	0.167	0.0118
7	0.437	0.201	0.0378	0.0303	0.015	0.0719	0.172	0.0120
8	0.439	0.201	0.0380	0.0312	0.0156	0.072	0.174	
9	0.442	0.203	0.0399	0.0337	0.0158	0.0725	0.175	
10	0.447	0.204	0.0418	0.0352	0.0169	0.0738	0.178	
11	0.452	0.205		0.0385	0.0182	0.0750	0.185	
12	0.454	0.210				0.0758		
Mean	0.437	0.200	0.0375	0.0300	0.0143	0.0717	0.167	0.0114
Std Dev	0.011	0.006	0.0021	0.0043	0.0025	0.0023	0.011	0.0005
C (95%)	0.007	0.004	0.0015	0.0029	0.0017	0.0015	0.007	0.0004

Note: $C_{(95\%)}$ is the 95% half-width confidence interval derived from the 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.

Participating Laboratories

ATI AllVac Ltd	Sheffield, England	UKAS accreditation 1385
Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
Metals Technology Testing Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Wu Han Steel Quality Inspection Station	WuHan, Hubei, China	CNAL accreditation 0721
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Pvt Ltd	Mumbai, India	NABL accreditation 0367
Genitest Inc	Montreal, Canada	
Coleshill Laboratories Ltd	Birmingham, England	
De Bruyn Spectroscopic Solutions	Western Cape, South Africa	
London & Scandinavian Met Co	Rotherham, England	

Note: to achieve National Accreditation (eg UKAS, NATA, A2LA, CNAL, NABL), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

Analytical Methods Used

ELEMENT	RESULT No. & METHOD				
	ICP-AES	XRF	FAAS		OTHER
Carbon	-	-	-	all	combustion (infra-red detection)
Silicon	4, 6-9	3	-	2, 5, 10, 11	gravimetric (perchloric acid)
Sulfur	-	-	-	1, 12	photometric (molybdenum blue)
Phosphorus	1, 3, 6, 8, 10, 11	-	-	all	combustion (infra-red detection)
Manganese	1, 3, 5, 8, 9	6	2	2, 4, 9	photometric (molybdenum blue)
Nickel	1-4, 6, 10	7	5, 9, 11-13	5, 7	volumetric (alkalimetric)
Chromium	1, 2, 5, 7, 9, 10	-	8, 11, 12	4, 10	photometric (periodate)
Molybdenum	1, 2, 5, 6, 9, 11	-	3, 7	7	volumetric (arsenite)
Copper	2, 4, 7-10	-	1, 3, 5, 11, 12	8	photometric (dimethyl glyoxime)
Cobalt	2, 4-6, 8, 9, 12	1	3, 7, 10, 11	3, 4, 6	volumetric (FAS)
Vanadium	1, 4, 6, 8-10	-	2, 5, 7	4, 8, 10, 12	photometric (thiocyanate)
Tungsten	1, 4-8, 10, 11	-	-	6	photometric (BCO)
Aluminium	2-5, 7, 8, 10	-	1, 9, 11	3	volumetric (FAS)
Titanium	2-5, 7, 10	-	1, 8	3, 9	volumetric (titanium chloride)
Tin	3, 4, 6-10	1	2, 5	2	ICP-MS
Arsenic	1-3, 7	-	4	6	photometric (chrome azurol S)
				6	ICP-MS
				9, 11, 12	photometric (di-antipyryl methane)
				11	photometric (phenyl fluorone)
				5	photometric (molybdenum blue)
				6	ICP-MS

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, 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 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 October 2027, 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.