

CERTIFICATE OF ANALYSIS

13X 12535 (batch BE)

Certified Reference Material Information

Type: STAINLESS STEEL (CHILL CAST)
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	Cu
Value ¹	0.229	1.407	0.0591	0.0400	0.342	14.79	16.95	4.09	0.130
Uncertainty ²	0.006	0.006	0.0015	0.0010	0.006	0.04	0.06	0.03	0.002

Element	Co	V	Sn	Al	Ti	Ta	B	N
Value ¹	0.146	0.252	0.0194	0.194	0.625	(0.020)	0.0051	0.029
Uncertainty ²	0.003	0.003	0.0006	0.010	0.012	-	0.0004	0.002

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

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 25th April 2016

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Method of Preparation

This reference material was produced from commercial-purity metals and master alloys. The discs are the product of one melt poured into a sequence of 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

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

Homogeneity

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Multiple measurements were taken from each surface under test.

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.

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

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 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.

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	0.217	1.390	0.0550	0.0392	0.327	14.73	16.83	4.029	0.1258
2	0.219	1.400	0.0553	0.0395	0.327	14.75	16.84	4.050	0.1260
3	0.220	1.401	0.0568	0.0397	0.328	14.75	16.87	4.052	0.1280
4	0.222	1.404	0.0572	0.0399	0.330	14.77	16.92	4.068	0.1283
5	0.228	1.405	0.0575	0.0400	0.337	14.79	16.94	4.068	0.1299
6	0.231	1.407	0.0583	0.0401	0.338	14.80	16.99	4.072	0.1300
7	0.232	1.411	0.0597	0.0401	0.345	14.81	17.01	4.095	0.1302
8	0.233	1.412	0.0600	0.0404	0.347	14.81	17.01	4.099	0.1304
9	0.237	1.417	0.0601	0.0408	0.348	14.81	17.02	4.117	0.1309
10	0.239	1.420	0.0606		0.349	14.83	17.03	4.118	0.1319
11	0.242		0.0607		0.351		17.03	4.125	0.1320
12			0.0608		0.351			4.134	0.1325
13			0.0616		0.353				0.1330
14			0.0632		0.356				
Mean	0.229	1.407	0.0591	0.0400	0.342	14.79	16.95	4.086	0.1299
Std Dev	0.009	0.009	0.0024	0.0005	0.011	0.03	0.08	0.034	0.0023
C (95%)	0.006	0.006	0.0014	0.0004	0.006	0.02	0.05	0.022	0.0014

Sample	Co	V	Sn	Al	Ti	Ta	B	N
1	0.1380	0.2428	0.0184	0.179	0.612	0.0175	0.0042	0.0245
2	0.1382	0.2436	0.0185	0.180	0.613	0.0183	0.0046	0.0250
3	0.1393	0.2460	0.0188	0.186	0.618	0.0201	0.0048	0.0279
4	0.1447	0.2476	0.0190	0.190	0.618	0.0203	0.0049	0.0290
5	0.1450	0.2510	0.0191	0.197	0.625	0.0208	0.0049	0.0293
6	0.1454	0.2520	0.0192	0.197	0.627	0.0210	0.0051	0.0305
7	0.1459	0.2520	0.0198	0.198	0.630	0.0225	0.0051	0.0320
8	0.1466	0.2530	0.0199	0.200	0.634		0.0052	0.0322
9	0.1473	0.2537	0.0199	0.200	0.635		0.0056	
10	0.1478	0.2540	0.0200	0.206	0.642		0.0062	
11	0.1485	0.2580	0.0209	0.206				
12	0.1487	0.2580						
13	0.1500	0.2585						
14	0.1508							
15	0.1540							
Mean	0.1460	0.2516	0.0194	0.194	0.625	0.0201	0.0051	0.0288
Std Dev	0.0046	0.0053	0.0008	0.009	0.010	0.0017	0.0005	0.0029
C (95%)	0.0025	0.0032	0.0005	0.006	0.007	0.0016	0.0004	0.0024

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

Exova Ltd.	Middlesbrough, UK	UKAS accreditation	0239
Sheffield Analytical Services	Sheffield, UK	UKAS accreditation	0012
Metals Technology (Testing) Ltd.	Sheffield, UK	UKAS accreditation	0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation	0492
Shanghai JinYi Test Technology Co. Ltd	Shanghai, China	CNAS accreditation	0783
Shandong Metallurgical & Science Research Bureau Veritas CPS Ltd.	Jinan, Shandong, China	CNAS accreditation	1461
TCR Engineering Services PVT. Ltd.	Chennai, India	NABL accreditation	0025
Raghavendra Spectro Metallurgical Laboratory	Mumbai, India	NABL accreditation	0367
Instytut Metalurgii Zelaza	Bangalore, India	NABL accreditation	0371
TEC Eurolab SRL	Gliwice, Poland	PCA accreditation	AB554
Mineral and Metallurgical Laboratories	Modena, Italy	ACCREDIA accreditation	52
AMG Superalloys UK Ltd	Bangalore, India		
Analyticka Laborator Lithea sro	Rotherham, UK		
Coleshill Laboratories Ltd	Brno, Czech Republic		
	Coleshill, UK		

Note: to achieve the above accreditation (UKAS, NATA, etc), test houses must 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	1, 5-7, 9	-	2, 3, 8 4, 10	gravimetric (perchloric acid) photometric (molybdenum blue)
Sulfur	8, 9	-	1-7, 10-14	combustion (IR or volumetric detection)
Phosphorus	1, 5, 6, 8, 9	-	2, 3, 7 4	photometric (molybdenum blue) volumetric (alkalimetric)
Manganese	1, 3, 4, 7, 8, 10-12	2, 5	6, 14 9, 13	photometric (periodate) volumetric (arsenite, FAS)
Nickel	4, 5, 8	1	2, 3, 6, 7, 9 10	gravimetric (dimethyl glyoxime) photometric (dimethyl glyoxime)
Chromium	1, 5, 8, 9	-	2-4, 6, 10, 11 7	volumetric (ferrous ammonium sulfate) photometric (diphenyl carbazide)
Molybdenum	4, 6, 8, 10-12	2, 5	1, 3, 7, 9	photometric (thiocyanate)
Copper	3-5, 7, 9, 10, 12	1, 6, 8	2, 11 13	photometric (bco) volumetric (thiosulfate)
Cobalt	2, 4, 6-8, 10-14	1, 3, 9	5 15	photometric (2beta-naphthol) gravimetric (oxide)
Vanadium	1, 3, 5, 6, 9, 11-13	2, 4, 10	7 8	volumetric (ferrous ammonium sulfate) volumetric (peroxi-di-sulphate)
Tin	2-6, 8-11	1, 7		
Aluminium	1, 2, 4, 6, 9, 10	3, 8, 11	5, 7	photometric (chrome azurol s)
Titanium	2, 3, 6, 8, 10	4, 5, 7	1, 9	photometric (diantipyryl methane, peroxide)
Tantalum	1-7	-		
Boron	1-10	-		
Nitrogen	-	-	1, 6 2, 3, 5, 7 4, 8	volumetric (hydrochloric acid) inert gas fusion (thermal conductivity) 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 31-2015 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 this method of 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 to a depth of 12mm. 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 April 2036, 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 L Maxim, Technical Director, MBH Analytical Ltd.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.