

# CERTIFICATE OF ANALYSIS

**13X 12548 (batch M)**

## Certified Reference Material Information

Type: RESULTURISED 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
Value <sup>1</sup>	0.189	0.421	0.218	0.0260	0.576	1.08	12.98
Uncertainty <sup>2</sup>	0.004	0.007	0.003	0.0013	0.011	0.02	0.08

Element	Mo	Cu	Co	Nb	W	Sb	N
Value <sup>1</sup>	1.33	0.228	0.352	0.585	0.033	0.022	0.0508
Uncertainty <sup>2</sup>	0.02	0.003	0.004	0.010	0.002	0.003	0.0017

## Definitions

- <sup>1</sup> 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.
- <sup>2</sup> 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 11th December 2011

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 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 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 - 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.

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
1	0.181	0.408	0.210	0.0220	0.558	1.043	12.81
2	0.185	0.411	0.213	0.0239	0.561	1.051	12.84
3	0.187	0.412	0.215	0.0244	0.564	1.056	12.90
4	0.187	0.414	0.217	0.0245	0.575	1.058	12.91
5	0.188	0.414	0.218	0.0261	0.577	1.067	12.96
6	0.189	0.423	0.219	0.0265	0.578	1.075	12.98
7	0.189	0.428	0.219	0.0268	0.580	1.082	13.06
8	0.191	0.431	0.220	0.0275	0.584	1.095	13.09
9	0.192	0.433	0.221	0.0276	0.592	1.113	13.11
10	0.195	0.436	0.225	0.0280	0.593	1.120	13.13
11	0.199		0.225	0.0283		1.128	
12						1.129	
<b>Mean</b>	<b>0.189</b>	<b>0.421</b>	<b>0.218</b>	<b>0.0260</b>	<b>0.576</b>	<b>1.084</b>	<b>12.98</b>
<b>Std Dev</b>	0.005	0.010	0.005	0.0020	0.012	0.031	0.11
<b>C<sub>(95%)</sub></b>	0.003	0.007	0.003	0.0013	0.009	0.020	0.08

Sample	Mo	Cu	Co	Nb	W	Sb	N
1	1.296	0.222	0.347	0.570	0.0303	0.0181	0.0480
2	1.313	0.225	0.347	0.570	0.0307	0.0188	0.0488
3	1.315	0.227	0.348	0.574	0.0308	0.0192	0.0491
4	1.315	0.227	0.351	0.586	0.0319	0.0193	0.0493
5	1.318	0.228	0.352	0.586	0.0325	0.0200	0.0500
6	1.343	0.228	0.353	0.593	0.0352	0.0208	0.0500
7	1.348	0.229	0.353	0.595	0.0352	0.0251	0.0510
8	1.350	0.230	0.353	0.596	0.0355	0.0258	0.0530
9	1.354	0.230	0.354	0.599		0.0277	0.0536
10	1.354	0.235	0.358				0.0550
11	1.368		0.361				
<b>Mean</b>	<b>1.334</b>	<b>0.228</b>	<b>0.352</b>	<b>0.585</b>	<b>0.0328</b>	<b>0.0216</b>	<b>0.0508</b>
<b>Std Dev</b>	0.023	0.003	0.004	0.011	0.0022	0.0036	0.0023
<b>C<sub>(95%)</sub></b>	0.016	0.002	0.003	0.009	0.0019	0.0027	0.0017

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 Materials Testing	Middlesbrough, England	UKAS accreditation 0239
IncoTest Ltd	Hereford, England	UKAS accreditation 0281
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Universal Scientific Laboratory	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation 0371
De Bruyn Spectroscopic Solutions	Johannesburg, South Africa	
Coleshill Laboratories Ltd	Birmingham, England	
London & Scandinavian Met. Co Ltd	Rotherham, England	

Note: to achieve the above-noted 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 (IR or volumetric detection)
Silicon	1, 4-6, 8	-	2, 3, 7 gravimetric (perchloric acid) 9, 10 photometric (molybdenum blue)
Sulfur	-	-	all combustion (IR or volumetric detection)
Phosphorus	1-3, 6, 7, 9, 11	-	4, 5, 8 photometric (molybdenum blue) 10 volumetric (alkalimetric)
Manganese	1, 8-10	2, 5	4, 6 photometric (periodate) 3, 7 volumetric (ferrous ammonium sulfate)
Nickel	1, 3-5, 8, 9, 11, 12	6	10 gravimetric (dimethyl glyoxime) 2, 7 photometric (dimethyl glyoxime)
Chromium	2, 4, 6, 10	-	1, 3, 5, 7-9 volumetric (ferrous ammonium sulfate)
Molybdenum	1-4, 6, 9, 11	7	5, 8, 10 photometric (thiocyanate)
Copper	1-6	7, 8	9, 10 volumetric (thiosulfate)
Cobalt	1-7, 9	8, 11	10 photometric (5 chloro-PADAB)
Niobium	1-4, 8, 9	6	5 gravimetric 7 photometric (chlorosulfophenol)
Tungsten	1, 2, 4-8	-	3 ICP-MS
Antimony	3, 4, 6, 8, 9	1, 2, 5	7 photometric (crystal violet)
Nitrogen	-	-	4 volumetric (hydrochloric acid) 10 photometric (Nessler reagent)
			1-3, 5-9 inert gas fusion (thermal conductivity)

## 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 semi-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. This certification will therefore expire in December 2031, 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.