

# CERTIFICATE OF ANALYSIS

**13X 15059 (batch P)**

## Certified Reference Material Information

Type: MARTENSITIC 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
Value <sup>1</sup>	<b>0.084</b>	<b>0.495</b>	<b>0.0133</b>	<b>0.0130</b>	<b>0.995</b>	<b>1.584</b>	<b>15.85</b>	<b>0.600</b>
Uncertainty <sup>2</sup>	0.002	0.008	0.0006	0.0008	0.012	0.013	0.06	0.005

Element	Cu	Co	Nb	V	W	Ti	N
Value <sup>1</sup>	<b>0.105</b>	<b>0.266</b>	<b>0.430</b>	<b>0.070</b>	<b>0.081</b>	<b>0.038</b>	<b>0.113</b>
Uncertainty <sup>2</sup>	0.002	0.005	0.006	0.002	0.002	0.002	0.004

## 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 17<sup>th</sup> December 2013

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	Mo
1	0.0803	0.478	0.0114	0.0111	0.971	1.551	15.76	0.588
2	0.0804	0.484	0.0117	0.0111	0.976	1.556	15.81	0.589
3	0.0833	0.486	0.0128	0.0114	0.977	1.557	15.82	0.591
4	0.0834	0.493	0.0129	0.0127	0.978	1.584	15.83	0.598
5	0.0835	0.493	0.0132	0.0129	0.980	1.585	15.83	0.598
6	0.0835	0.493	0.0132	0.0130	0.985	1.586	15.83	0.599
7	0.0837	0.500	0.0135	0.0131	1.000	1.586	15.84	0.600
8	0.0846	0.502	0.0135	0.0131	1.007	1.587	15.85	0.600
9	0.0849	0.512	0.0136	0.0132	1.010	1.600	15.86	0.601
10	0.0850	0.512	0.0139	0.0141	1.013	1.603	15.91	0.602
11	0.0852		0.0141	0.0145	1.020	1.606	15.93	0.604
12	0.0856		0.0146	0.0146	1.020	1.608	15.98	0.607
13			0.0148	0.0146				0.609
14								0.617
<b>Mean</b>	<b>0.0836</b>	<b>0.495</b>	<b>0.0133</b>	<b>0.0130</b>	<b>0.995</b>	<b>1.584</b>	<b>15.85</b>	<b>0.600</b>
<b>Std Dev</b>	0.0017	0.011	0.0010	0.0012	0.019	0.020	0.06	0.008
<b>C (95%)</b>	0.0011	0.008	0.0006	0.0008	0.012	0.013	0.04	0.005

Sample	Cu	Co	Nb	V	W	Ti	N
1	0.102	0.255	0.419	0.0652	0.0781	0.0336	0.105
2	0.102	0.255	0.423	0.0657	0.0790	0.0349	0.106
3	0.103	0.257	0.424	0.0682	0.0794	0.0350	0.108
4	0.104	0.258	0.425	0.0683	0.0795	0.0353	0.111
5	0.104	0.259	0.426	0.0684	0.0798	0.0358	0.112
6	0.104	0.260	0.427	0.0699	0.0804	0.0368	0.114
7	0.105	0.268	0.427	0.0702	0.0809	0.0385	0.117
8	0.106	0.268	0.430	0.0702	0.0811	0.0387	0.119
9	0.107	0.270	0.431	0.0713	0.0824	0.0395	0.119
10	0.110	0.271	0.435	0.0727	0.0825	0.0398	0.121
11	0.112	0.271	0.444	0.0735	0.0828	0.0400	
12		0.275	0.445	0.0738	0.0834	0.0400	
13		0.278				0.0416	
14		0.281				0.0421	
<b>Mean</b>	<b>0.105</b>	<b>0.266</b>	<b>0.430</b>	<b>0.0698</b>	<b>0.0808</b>	<b>0.0380</b>	<b>0.113</b>
<b>Std Dev</b>	0.003	0.009	0.008	0.0028	0.0017	0.0027	0.006
<b>C (95%)</b>	0.002	0.005	0.005	0.0018	0.0011	0.0016	0.004

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, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 492
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Bureau Veritas CPS Pvt	Chennai, India	NABL accreditation 0025
TCR Engineering Servs Pvt Ltd	Mumbai, India	NABL accreditation T367
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Genitest Inc	Montreal, Canada	PRI accreditation 123077
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
London & Scandinavian Met Co	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (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	3, 5-10	-	1, 2, 4	photometric (molybdenum blue)
Sulfur	2	-	1, 3-13	combustion (IR or volumetric detection)
Phosphorus	2, 6-9, 12, 13	-	1, 10, 11	volumetric (alkalimetric)
			3-5	photometric (molybdenum blue)
Manganese	2, 3, 5-7, 9, 11	1, 12	4, 10	photometric (periodate)
			8	volumetric (arsenite, FAS)
Nickel	1, 3, 5, 6, 9-12	-	2, 7	photometric (dimethyl glyoxime)
			4, 8	gravimetric (dimethyl glyoxime)
Chromium	1-3, 6-8, 12	-	4, 5, 9-11	volumetric (ferrous ammonium sulfate)
Molybdenum	1-3, 6-8, 10-13	4, 9	5, 14	photometric (thiocyanate)
Copper	1-3, 6, 7, 9-11	5, 8	4	photometric (BCO)
Cobalt	2, 4-9, 11, 12, 14	1, 3, 10	13	volumetric (iodine)
Niobium	1-3, 5-8, 10-12	-	4	ICP-MS
			9	photometric (chlorosulfophenol)
Vanadium	1, 3-12	2		
Tungsten	1, 2, 4-7, 9, 11, 12	8	3	photometric (thiocyanate)
			10	ICP-MS
Titanium	2, 3, 5, 6, 8-13	1, 7	4	ICP-MS
			14	photometric (di-antipyryl methane)
Nitrogen	-	-	3-9	inert gas fusion (thermal conductivity)
			2, 10	volumetric (hydrochloric acid)
			1	photometric (nessler's reagent)

## Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 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. Technical support for this certification will therefore expire in December 2033, 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.